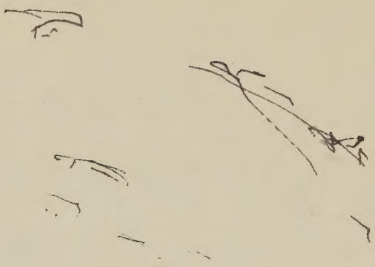

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THE TECHNICAL EXAMINATION OF CRUDE PETROLEUM, PETROLEUM PRODUCTS AND NATURAL GAS

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THE TECHNICAL EXAMINATION OF CRUDE PETROLEUM, PETROLEUM PRODUCTS AND NATURAL GAS

Including also the Procedures Employed in the Evaluation of Oil-Shale and the Laboratory Methods in Use in the Control of the Operation of Benzol-Recovery Plants.

BY

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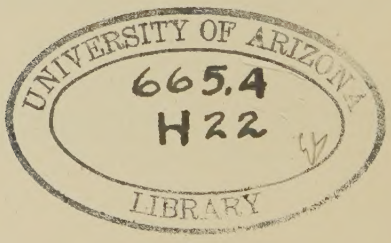

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TO THE MEMORY OF

ROBERT KENNEDY DUNCAN

Formulator of the Industrial Fellowship System and First Director of the Mellon Institute of Industrial Research of the University of Pittsburgh; Enthusiastic Advocate of Orderly Synthesis in Scientific Inquiries of Technologic Concernment, and Classic Interpreter of the Trend and Goal of the Sciences.

33935

PREFACE

This manual on analytical bitumenology has been prepared primarily to meet the requirements of students of hydrocarbon chemistry and petroleum engineering. There is, however, a generally recognized need in the chemical profession for a treatise which shall present the methods now in use for the technical examination and evaluation of the hydrocarbon complexes, natural gas, crude petroleum and oil-shale and their commercially important products; and the hope is expressed by the authors that this concise work will serve a useful purpose by making readily available to chemists and to engineers the procedures of applying the physical and chemical tests which are recognized as essential by technologists. Because of the significance of benzol and of benzol-gasoline mixtures as motor fuels, a chapter is devoted to the laboratory methods in use in the control of the operation of benzol-recovery plants. The *Appendix* contains in compact form chemical and physical data for the reference of the refinery chemist, petroleum engineer and gas engineer.

The first eight Chapters of the book supplement Chapters IV, XI, XII (**2,** 517–527, in particular), and XVII of Bacon and Hamor's "American Petroleum Industry," and the manual may be used, in fact, as a laboratory companion for that set. The authors request the indulgence of the user for any errors or omissions that occur. In a work of this character, relating mainly to the chemical control of the products of several industries which cover large and progressive fields, an early revision will probably be desirable, and the authors will therefore be glad to receive any suggestions with this end in view which may be submitted to them.

In the use of this treatise there is assumed a familiarity with the usual operations of volumetric and gravimetric analysis and some proficiency in organic chemistry. No list of the abbreviations of the titles of the technical periodicals cited has been prepared for the reason that the standard journal list of the American Chemical Society has been complied with in the frequent foot references to authorities and sources of information. The litera-

ture of the subjects treated is voluminous, which has necessitated judicious selection in the preparation of the bibliographic notes.

It is a pleasure for the authors to acknowledge their indebtedness to Mr. George H. Taber, Vice-President of the Gulf Refining Company, Pittsburgh, Pa., for his helpful advice. Mr. Taber has read the entire manuscript of the book and from his profound knowledge of naphthology has made many valuable criticisms, resulting in the improvement of the text throughout the work. The authors are also grateful to Dr. W. F. Faragher, of the Mellon Institute of Industrial Research, for his careful examination of the manuscript. Dr. Faragher's familiarity with hydrocarbon chemistry has enabled him to render numerous suggestions of importance in the preparation of the text for publication. The coöperative assistance given by Messrs. H. R. Auerswald, of the Gypsy Oil Company, W. E. Espy, of Cosden and Company, R. O. Neal, of the Bureau of Mines, and F. W. Sperr, Jr., of The Koppers Company, has likewise facilitated the undertaking of the authors and is highly appreciated by them.

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PITTSBURGH, PA.,
AND
NORMAN, OKLA.,
September, 1920.

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THE TECHNICAL EXAMINATION OF CRUDE PETROLEUM, PETROLEUM PRODUCTS AND NATURAL GAS

CHAPTER I

THE EXAMINATION AND EVALUATION OF CRUDE PETROLEUM

I.—The Examination of Crude Petroleum.

1. Specific Gravity and Baumé Gravity.
2. Color, Fluorescence and Odor.
3. Viscosity.
4. Water and B.S.
5. Heat of Combustion.
6. Sulphur.
7. Nitrogen and Oxygen.
8. Carbon and Hydrogen.
9. Index of Refraction.
10. Coefficient of Expansion; Specific Heat, and Heat of Vaporization.
11. Distillation:
    Engler-Ubbelohde Method;
    Bureau of Mines Methods: Rittman-Dean and Allen-Jacobs Methods.
12. Estimation of Paraffin Wax.
13. Estimation of Asphaltene.
14. Other Tests concerning Crude Petroleum:
    Formolite Number; Iodine Number, and Loss to Sulphuric Acid; the Determination of the Composition of Petroleum.

II.—The Commercial Evaluation of Crude Petroleum.

1. Preliminary Examination.
2. Final Examination:
   (A) Paraffin-Base; (B) Mixed-Base; (C) Asphalt-Base; (D) Pressure Distillation.
3. Notes and Practical Suggestions.
4. Theory of Refining.

The physical and chemical operations involved in the processes of refining crude petroleum are sufficiently understood that a quite effective control of the character of the products is possible,

and there is maintained at every large refinery a laboratory equipped for the systematic examination of the raw materials and their products.[1] From the analytical data in conjunction with physical tests, the details of the refinery procedures are modified, aiming at the largest output of products best suited for the specific purposes intended, and at the lowest cost. Analytical hydrocarbon chemistry is an indispensable aid to petroleum technology.

One of the most important duties of the refinery chemist is to examine and value a crude petroleum received at the plant or under consideration for purchase, and to decide as to its suitability and whether it is adapted for treatment by the usual processes or whether modifications are necessary. In the preparation of the present Chapter, the requirements of the refinery chemist are kept mainly in view, but an endeavor also is made to deal with the general nature of the several varieties of crude petroleum and to present such methods of examination as are likely to be needed by the consulting analyst or by the student of chemistry who wishes to determine the proximate components of or possible yield of commercial products from crude petroleum of any type. The various tests and procedures are arranged seriately, in the order followed in general practice.

I. THE EXAMINATION OF CRUDE PETROLEUM

1. Specific Gravity and Baumé Gravity.—The specific gravity may be ascertained by any of the methods described under gasoline (see page 41), the pycnometric method being preferable for accuracy, and the hydrometric for rapidity and convenience. The final result is reported in Baumé degrees (modulus 140) at 60°F. (see page 42).

When the product is too viscous for the hydrometer to be used and a pycnometer is not available, the following method is recommended: A drop of the oil to be tested is placed in a solution of ethyl alcohol and water, of approximately the same specific gravity as the oil. In case the oil sinks to the bottom of the receptacle, more water is added to the alcohol-water mixture. If the drop rises to the surface, more alcohol is added. This procedure is carried out until the drop of oil neither rises nor sinks in the liquid. The gravity of the liquid then is determined

[1] For a description of a modern petroleum research and control laboratory, see *Chem. Age* (Am.), **1** (1919), 85–9.

by a Baumé hydrometer and corrected to 60°F. This value is the Baumé gravity[1] of the oil.

2. Color, Fluorescence (Bloom) and Odor.—These characteristics may be determined from a sample placed in an ordinary (4-oz.) oil-sample bottle.[2] In order to define the *color*, the bottle is held between the eye and the light (transmitted light), looking through the length of the bottle when feasible. Where the oil under examination is too opaque for this observation, even with the most powerful light available, the determination can be made by observing the oil diametrically through the body of the bottle, or, that failing, diametrically through its neck. The color may vary from a yellow or yellowish red to an opaque black or greenish black. The fluorescence, or "bloom," is observed clearly by having the eye between the sample and the light (reflected light), the sample being held against a dark surface if necessary. Certain crudes of Texas exhibit a blue fluorescence, although this property generally is regarded as one characteristic of certain fractions rather than of crude petroleum itself.

The *odors* of different crude oils vary considerably. Pure paraffin-base crude petroleum having a high percentage of volatile constituents, will possess the distinctive odor of its contained gasoline. Crude oils of California, Texas, Rumania, Russia, and certain other localities, frequently have an aromatic odor, due to the presence of hydrocarbons of the benzene series. In order to determine the odor of crude petroleum, two sample bottles should be half-filled with the oil under examination, and then carefully

[1] Taber (*J. Ind. Eng. Chem.*, **12** (1920), 593) has considered some frequently misunderstood points respecting the Baumé hydrometer. Oftentimes a would-be purist in nomenclature who approaches the petroleum business from a scientific viewpoint, condemns the oil man for using the expression "Baumé gravity" and endeavors to pursuade him to say "Baumé specific gravity," which is more cumbersome and further from being correct. As Taber indicates, if time in speaking and time and space in writing were of no importance, we might be absolutely precise and say, for instance, in using the light Baumé scale: "The oil is of a specific gravity equivalent to 50° Baumé." This is, however, unnecessary and the expression "The oil is 50° Baumé gravity" cannot be misunderstood and is entirely justifiable. This subject is discussed at length by Taber, to whose contribution the reader is referred for particulars.

[2] The ordinary 4-oz. sample bottle used in the oil business is about 1½-in. in outside diameter by 5½-in. in length, with a neck 0.8-in. in diameter by ¾-in. in length. When used for the taking of color, it preferably has a ground bottom.

stoppered. One of the bottles is shaken vigorously, so as to impart the odor to the air above the oil. If the odor of hydrogen sulphide is detected, 5 c.c. of a concentrated solution of sodium or potassium hydroxide should be added, and the oil shaken until the odor of hydrogen sulphide disappears. In the case of a number of crude oils from California, agitation of the oil with alkali solution will develop the odor of pyridine. In the second sample, the odor should be noted after similar treatment with 5 c.c. of dilute (5N) sulphuric acid.

3. Viscosity.—Crude petroleums vary in consistency from the thin oils of the Appalachian field to the extremely viscous and even solid products of Mexico and California. Suitable methods for determining the viscosity may be selected from those described under illuminating oils (page 84), lubricating oils (page 90), or asphalt (pages 129 and 157).

4. Water and B.S.—In order to be salable, or classed as "pipe line oil" and accepted by the transporting companies, a crude petroleum must contain generally no more than 1 per cent. of water and B.S. This necessitates some treatment of emulsions in the field.[1] The amount of water in oil-field emulsions varies from traces up to 60 per cent. or more, but the majority of emulsions encountered in the Mid-Continent and Gulf fields contain about 25 per cent. The sediments ("Bottom Settlings," or "B.S.") accumulating in tanks usually contain about 50 per cent. of water.

The rapid method of determining the combined water and B.S. is to dilute the crude petroleum with an equal volume of gasoline or of other suitable solvent, and then to centrifuge in a vessel constricted at the lower end, so that more accurate readings of the water and B.S. may be made (see Fig. 1). The operation is continued until all sediment has been thrown to the bottom—about 20 minutes. The volume of the water and B.S. accumulated in the constricted portion of the graduate finally is read, and the percentage by volume of the water and B.S. in the original crude is calculated therefrom. The Goetz tube gives very satisfactory results in this test.[2]

[1] See Sherrick, *J. Ind. Eng. Chem.*, **12** (1920), 133.

[2] The following method is used for determining water and B.S. in Mexican crude petroleum: A mixture is made of 25 c.c. of carbon disulphide, 25 c.c. of gasoline, and 50 c.c. of the oil under test. The mixture is agitated thoroughly and revolved in a centrifuge at room temperature, at the rate of about 1,500 revolutions per minute, until the water and B.S. separate shows no increase or decrease.

An accurate determination of the water in petroleum may be made by the following method:

Apparatus and Reagents.—250-c.c. flask, with a neck of medium length; special graduate for receiving the distillate (see Fig. 2); asbestos paper for wrapping the neck of the flask; condenser; sand-bath; and bunsen burner.

Procedure.—The neck and upper part of the flask are covered with asbestos paper, and 200 c.c. of the oil are measured out and

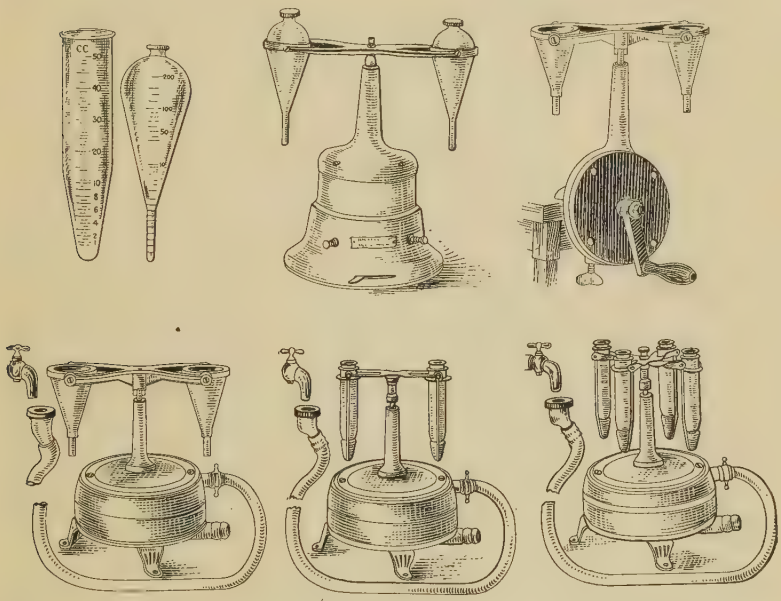

FIG. 1.—Electric, hand-power and water-power centrifuges. The hand-power centrifuge is provided with the Goetz attachment, which allows the use of 100-c.c. glass tubes.

weighed into the flask, which is placed in a sand-bath and connected with the condenser. The special graduate is located at the outlet of the condenser. It is well to place a few small fragments of porous plate in the flask with the oil, to assist in preventing bumping. Finally, the flame is started under the sand-bath and the distillation is begun. The distillation is carried on rapidly until all the water has come over. The complete removal of water is indicated by regular ebullition after all "bumping" has ceased. In order to get all the water into the aqueous layer at the bottom of the graduate, a few drops of a concentrated solution of asphalt in benzene are added to the

distillate, and the cylinder is tapped until the drops of water observed on the sides against the darkened oil layer have settled to the bottom.[1] The volume of the water at the bottom of the cylinder is noted, and from this datum and the weight of the oil previously taken the percentage by weight of water in the original crude is calculated.

FIG. 2.—Graefe's oil cylinder, graduated in c.c., for determining water in fuel oil and crude petroleum. In addition to its use as a receiver, this cylinder may be, and, in fact, often is, employed in the determination of water and B. S. by settling, in cases where a centrifuge is not convenient. A separatory funnel, with stopcock, capacity 120 c.c., with outlet tube graduated in $\frac{1}{5}$-c.c. divisions, also may be used for both purposes.

Certain heavy crudes which are badly emulsified[2] are very difficult to distil by the above method. In such cases it is recommended that the crude oil be diluted with solvent gasoline, benzene, toluene, or a mixture of 80 per cent. of xylene and 20 per cent. of benzene before the distillation is begun.[3]

The distillation method of Allen and Jacobs is especially adapted to dealing with crude petroleums containing water. This method is described on page 17. The bulb and neck of the flask are covered with an insulating jacket, in which is imbedded the resistance wire which is the source of heat. The vapors coming over are held at the same temperature as the oil by means of a shunt-circuit about the neck of the flask. This provision obviates the condensation of water in the neck of the flask and hence all bumping due to the reflux of water into the body of the oil.

5. Heat of Combustion.—The most reliable method for the determination of the heat of

[1] See FUCHS, *Eng. Min. J.*, **106** (1918), 357.

[2] On emulsified oil and its properties, see MCCOY, SHIDEL and TRAGER, *Bull. Am. Inst. Min. Met. Eng.*, August, **1919**, 1513. On the modern conception of emulsions, see CLAYTON, *J. Soc. Chem. Ind.*, **38** (1919), 113T. On the properties and treatment of oil-field emulsions, see SHERRICK, *J. Ind. Eng. Chem.*, **12** (1920), 133. See also GLASER, *Feuerungstech.*, **18** (1920), 149.

[3] On the determination of water in petroleum, see DEAN and STARK, *J. Ind. Eng. Chem.*, **12** (1920), 486. According to these authors, the method of distilling in the presence of an immiscible solvent is the most generally satisfactory scheme for estimating the water content of petroleum emulsions; they have developed modifications of this method which add to its convenience.

combustion of crude petroleum is that of combustion in a bomb with oxygen at 30 to 40 atmospheres pressure.[1] For the details of this method, see Poole's "The Calorific Power of Fuels," Brame's "Fuel, Solid, Liquid and Gaseous," Stillman's "Engineering Chemistry," or Holde-Mueller's "Examination of Hydrocarbon Oils."

Where it is not convenient actually to determine the calorimetric value this quantity may be estimated approximately by means of the following equation:[2] Calorific value in B.t.u. = 18,650 + 40 (Baumé degrees − 10).

6. Sulphur.—Sulphur may be determined conveniently and accurately during the combustion in a bomb with oxygen, and, if desired, at the same time as the calorimetric determination mentioned above. The following procedure is from *Technical Paper* **26** of the United States Bureau of Mines:

A portion of a sample, 0.7 to 1.0 g., is burned in a calorimetric bomb containing 10 c.c. of water and oxygen under a pressure of 30 atmospheres. A lower pressure sometimes gives inaccurate results. If the sample contains more than 3 per cent. of sulphur, the bomb is allowed to stand in its water-bath for 15 minutes after ignition of the charge. In case the sulphur content is as high as 5 per cent., oxygen under 40 atmospheres pressure is used. With these high pressures in a Berthelot bomb of 500-c.c. to 600-c.c. capacity, repeated trials have failed to show even traces of carbon monoxide or sulphur dioxide. If a smaller bomb of about 175-c.c. capacity, such as that of Peters or of Kröker, is used, incomplete combustion from a lack of oxygen may result, if too large a sample is taken.

After cooling—15 minutes is usually enough—the bomb is opened and its contents are washed into a beaker. If the bomb has a lead washer, 5 c.c. of a saturated solution of sodium carbonate are added, the contents are heated to the boiling-point, boiled for 10 minutes, and are then filtered. This operation is necessary to decompose any lead sulphate from the washer. The united washings are then filtered, acidified with hydrochloric acid, boiled to expel all carbon dioxide, and the sulphuric acid is determined in the usual way with barium chloride.

Gravimetric determination is preferred to volumetric, because the nitrogen contained in the air originally in the bomb is oxidized in part

[1] Some difficulty in weighing occasionally is encountered when calorific value determinations are made on petroleum samples which contain very volatile hydrocarbons. In laboratories where a considerable number of these determinations are made the device described by Foster (*Chemist-Analyst*, **1917**, No. 21, 23) will permit samples to be weighed with minimum loss due to evaporation.

[2] Sherman and Kropff, *J. Am. Chem. Soc.*, **30** (1908), 1626.

to nitro-acids, which cause a small error if the volumetric determination alone is used. The sulphur content of any combustible material, from light gasolines weighed in a tared gelatin capsule, to solid bitumens and cokes, can be readily determined by this method.

This method of burning in a bomb is accurate, practicable, and rapid, and is recommended in preference to all other procedures.

Sulphur may also be determined in crude petroleum by the method of Carius, wherein the crude oil in a bomb-tube is oxidized by means of fuming nitric acid (the details of this method are presented in the standard works on organic analysis[1]). The lamp method is sometimes used in determining sulphur in crude petroleum (see pages 59, 75 and 97): the oil is burned in a lamp and the products of combustion are absorbed in standard sodium carbonate or sodium hypobromite solution. In the former case, the sodium carbonate is titrated with standard sulphuric acid and in the latter case the sulphate ion is precipitated in the usual way.

A qualitative test for sulphur which is reliable, and which, with practice, may be used as a rough means of quantitative estimation, is as follows:[2]

[1] The manipulation of the Carius method is described in detail in Gattermann's "Practical Methods of Organic Chemistry," 3rd ed. For a consideration of methods, see *Tech. Paper* **26**, Bureau of Mines, 1912. See also Regester, *J. Ind. Eng. Chem.*, **6** (1914), 812; and Christie and Bisson, *ibid.*, **12** (1920), 171.

The fusion method is used by some chemists in determining sulphur in crude petroleum. For this purpose the latest Parr bomb is recommended, using 10 g. of sodium peroxide, 1 g. of potassium chlorate, and 0.2 g. of benzoic acid, with 0.2 g. of the oil. During the manipulation a blank should be run on the chemicals used. Francis and Crawford (*J. Ind. Eng. Chem.*, **9** (1917), 479) report good results with the Parr bomb on Cushing oil, although the experts of the U. S. Bureau of Mines (*Tech. Paper* **26**) do not look with so much favor on the fusion method as applied to petroleum.

On the presence of sulphur and sulphur compounds in petroleum, see Mabery and Smith, *Am. Chem. J.*, **13** (1891), 233; Mabery and Quayle, *Proc. Am. Acad.*, **41** (1905), 89; Peckham, *Proc. Am. Phil. Soc.*, **36** (1897), 108; Richardson and Wallace, *Eng. Min. J.*, **73** (1902), 352; and Thiele, *Chem.-Ztg.*, **26** (1902), 896.

[2] Francis and Crawford (*J. Ind. Eng. Chem.*, **9** (1917), 479) favor the use of the methylene blue test for the detection of small amounts of sulphur. However, this test is more difficult of manipulation than the nitroprusside test. The procedure is as follows: The oil is heated with metallic sodium in the usual way and the filtered solution is neutralized in the presence of a drop or two of phenolphthalein solution, then 5 or 6 drops of hydrochloric acid are added in excess. A small crystal of p-amido dimethylaniline sul-

Apparatus and Reagents.—Metallic sodium; test-tube; small beaker; filter paper; small glass funnel; a 3 per cent. solution of sodium nitroprusside; and distilled water.

Procedure.—Two c.c. of the oil are decomposed by heating in a test-tube with about 0.25 g. of sodium. While still hot, the tube is broken by plunging into 8 c.c. of distilled water in a small beaker, or, better still, in a small casserole. Any particles of sodium surrounded by coke or tar are broken up so that the sodium is consumed. The solution then is filtered and tested with a few drops of the solution of sodium nitroprusside. A violet color indicates the presence of sulphur.

The "doctor" or plumbite test for petroleum distillates is described on page 59.

7. Nitrogen and Oxygen.—*Nitrogen* is present in crude petroleum generally in the form of derivatives of pyridine and quinoline.[1] If a large volume of the crude oil is shaken with a small volume of dilute (5N) sulphuric acid, and the acid then is separated and made alkaline with sodium hydroxide, the bases are set free and may be extracted with ethyl ether and subsequently investigated. The odor of pyridine bases may often be recognized in the alkaline solution mentioned.

Nitrogen may be detected in a crude oil when testing for sulphur. To a portion of the filtered solution which, in the case of sulphur, is to be tested with sodium nitroprusside, a solution of a ferrous salt is added, then a solution of a ferric salt, the mixture is boiled for a minute, and finally sufficient hydrochloric acid is added to make the solution acid in reaction. A blue precipitate or a blue or a green color indicates the presence of nitrogen.

The method of Kjeldahl[2] is recommended for the quantitative

---

phate is dissolved in the liquid and several drops of a dilute ferric chloride solution are added. If the oil contains sulphur, a blue color is developed which becomes more intense on standing.

[1] See PECKHAM, *Am. J. Sci.*, (3), **48** (1894), 250; and MABERY, *J. Soc. Chem. Ind.*, **19** (1900), 505. The recent investigations of MABERY (*J. Am. Chem. Soc.*, **41** (1919), 1690) have shown that all crude petroleums contain at least traces of nitrogen compounds.

[2] In the use of the Kjeldahl method it is probable that the best results are secured under the following conditions: The weight of the sample may be as much as 5 g. for crude oils low in nitrogen. The use of 25 c.c. of conc. c.p. sulphuric acid, 10 g. of potassium sulphate, 0.7 g. of mercury or mercuric oxide, and a time of heating of 2½ hrs., are recommended. In addition, a condenser placed in the neck of the flask is advantageous.

determination of nitrogen in petroleum. It is of interest to mention, however, that Mabery has developed a method which is a combination of the Dumas procedure for nitrogen and the oxygen combustion for carbon; it is said to be applicable to petroleums containing but minute amounts of nitrogen.

Oxygen exists in crude petroleum usually in the form of naphthenic acids or phenol derivatives which are soluble in strong alkali.[1] If a large quantity of the crude oil is agitated with a small amount of concentrated sodium hydroxide solution, and the alkaline solution is separated and acidified, the naphthenic acids may be differentiated from the phenolic bodies by the odor. The phenols have a creosote-like odor, while the crude naphthenic acids, especially upon warming, possess a suilline cacodor.[2]

The percentage of oxygen in petroleum is estimated by subtracting from 100 the sum of the percentages of the other elements present. The combined errors in the determination of the other elements will thus find expression in the oxygen content of the crude oil. For this reason, and because of the fact that sulphur has been ignored in the analysis of some crude petroleums, it is likely that the literature consequently contains many data which are misinforming.

8. Carbon and Hydrogen.—The carbon and hydrogen contents of different crude oils will, of course, vary with the nature of the oils. Thus, straight paraffin-base crude petroleum will have relatively the most hydrogen, while the dense asphaltic crudes will contain relatively the least.

The method used for the quantitative determination of carbon and hydrogen is the very satisfactory one of ultimate analysis, in which the hydrocarbons are burned to water and carbon dioxide in a stream of oxygen in the presence of cupric oxide. The method is described at length in Benedict's "Elementary Organic Analysis," Clarke's "Handbook of Organic Analysis," Hans Meyer's "Analyse und Konstitutionsermittlung Organischer Verbindungen," Lassar-Cohn's "Arbeitsmethoden," and other standard works. When sulphur is known to be present, a layer of lead peroxide is placed in the tube next to the absorption

[1] See Mabery, *J. Am. Chem. Soc.*, **28** (1906), 596. See also Bacon and Hamor's "American Petroleum Industry," 1916, **1,** 7; **2,** 581, 620, 803 and 888.

[2] On the malodor of naphthenic acids, see *Petroleum*, **9** (1914), 1506; and *Mat. grasses*, **7** (1914), 4115.

apparatus; while if nitrogen is present, a freshly reduced spiral of copper gauze is inserted in the tube beyond the copper oxide.[1]

9. Index of Refraction.—The refractive index, determined for the crude oil and certain of its fractions, along with specific gravity and range of boiling-point, may be of value in indicating the locality from which the petroleum is obtained.[2]

The instrument of Abbé is well adapted for the determination of the index of refraction; this refractometer has been described so often that an account of it is unnecessary here, and the reader is referred to Lewkowitsch's "Technology of Oils, Fats and Waxes," Leach's "Food Analysis," or Holde-Mueller's "Examination of Hydrocarbon Oils."

The correction to be applied to this instrument for each degree centigrade is, on the average, 0.0004; that is, for each rise in temperature of one degree centigrade, the index of refraction becomes less by that amount. The standard temperature for the index of refraction is 60°F. and all values should be corrected to that temperature.

10. Coefficient of Expansion; Specific Heat, and Heat of Vaporization.—*Coefficient of Expansion.*—The United States Bureau of Standards has carried out an extensive research on the thermal density coefficients of American petroleum. For a description of the methods used and data leading to the computation of correction tables, consult *Technologic Paper* **77** of the Bureau of Standards. The correction tables may be found on page 304 of this book. Other methods for the determination of the coefficient of expansion are described in Bacon and Hamor's "American Petroleum Industry" (**1,** 96) and in Holde-Mueller's "Examination of Hydrocarbon Oils."

Specific Heat.—An average value, sufficiently close for some engineering calculations, is about 0.45. Detailed methods of determination are given in Holde-Mueller's "Examination of Hydrocarbon Oils" and in Bacon and Hamor's "American Petroleum Industry" (**1,** 104).

Heat of Vaporization.—A curve showing the average change of heat of vaporization with boiling-point, may be found in the *Appendix,* page 325. Values taken from this curve may be used in certain engineering calculations. For methods of determining

[1] See *Bulletin* **46,** Bureau of Chemistry, U. S. Dept. of Agric.

[2] RITTMAN, *J. Ind. Eng. Chem.,* **7** (1915), 578.

the latent heat of evaporation, see Holde-Mueller's "Examination of Hydrocarbon Oils."

11. Distillation.—The original Engler method of distillation involved the use of the flask shown in Fig. 3 and a sample of 100 c.c. When the temperature of the vapor at the side-arm reached 150°C. (302°F.), the flame was withdrawn and the temperature was allowed to drop 20°C. (36°F.), when the heat was again applied until 150°C. was reached. This procedure was repeated until no more distillate came over below 150°C. The distillate thus secured was designated the gasoline or naphtha fraction. The heating then was continued until a temperature of 300°C. (572°F.) was attained, when the flame was withdrawn and the temperature allowed to drop as before. The distillate thus secured was described as the kerosene or illuminating oil fraction. The residue in the flask represented the lubricating oil fraction, and contained the paraffin wax and the asphaltene, provided the crude was of such a nature that the two last named constituents were present.

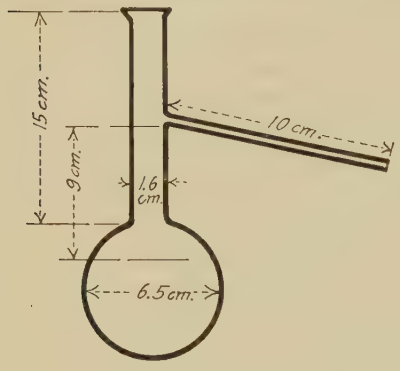

FIG. 3.—Engler distillation flask.
(See also page 54.)

The Engler-Ubbelohde Method.—This method is similar to the Engler method, except that temperature drops are prevented during the distillation. Many of the analyses of crude oils appearing in the literature have been conducted in accordance with this method, and it is still very extensively used.[1]

The following description of the method contains certain additional manipulations, which are included for the purpose of securing supplemental data.

Apparatus.—Standard Engler flask; distillation thermometer reading from a temperature of 40°C. to at least 300°C. in 1-degree divisions; auxiliary thermometer (to be used in case the distillation thermometer has been calibrated for total immersion), upon which temperatures from 15°C. to 50°C. may be read; condenser

[1] See *Mineral Resources of the United States*, **1913**, ii, 1125.

having an inner tube 60 cm. long; burner of such design that the height of flame may be slowly and continuously increased (see page 51); shield for burner and bulb of flask;[1] and five receiving vessels (25 c.c. graduated cylinders or a Brühl receiver containing 5 graduated tubes may be used).

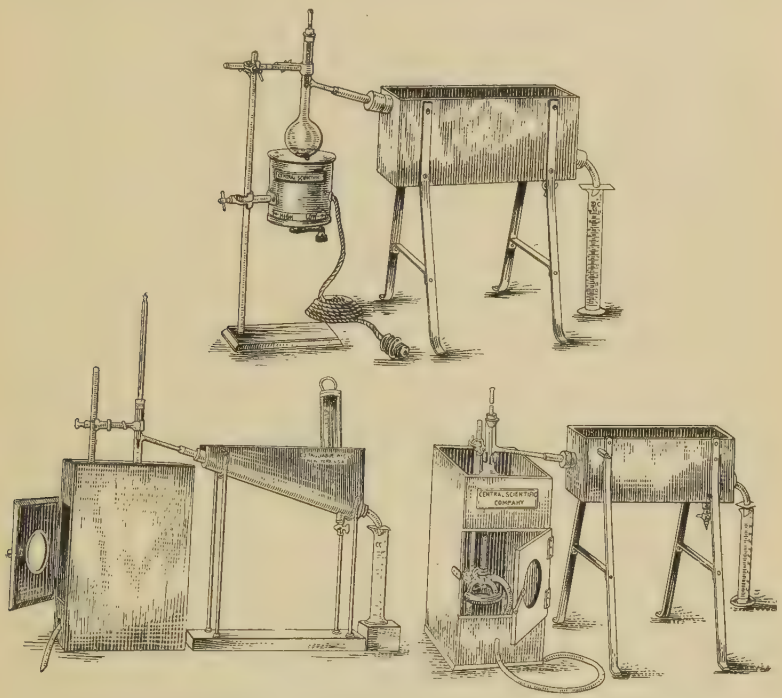

FIG. 4.—Distillation apparatus offered by laboratory supply houses.

Procedure.—100 c.c. of the oil are measured out at a temperature of 60°F. and weighed into the flask.[2] The distillation

[1] Laboratory supply houses offer distillation apparatus which includes a tank condenser and a shield for the flask and burner (see Fig. 4).

[2] Larger samples may be, and often are, distilled analytically in flasks of the Engler type. In this way, 200, 500 and 1000 c.c. samples are treated, with the advantage that larger fractions are secured, the gravities of which may be determined in the receivers by means of a small hydrometer. The larger the sample distilled, the lower the over-point. The analytical procedure is varied according to refining practice. To illustrate, if it is the practice to refine *in toto*, the laboratory procedure involves the separation of a complete line of crude products. If, however, the crude oil is handled in a skimming plant, the analysis merely includes the separation of crude naphtha, crude lamp distillate, gas oil, and bottoms.

thermometer and cork are inserted so that the upper portion of the thermometer bulb is opposite the lower side of the outlet tube. The outlet tube from the flask is attached to the condenser tube by means of a well-rolled cork, and one of the receivers is placed in position at the outlet of the condenser. The auxiliary thermometer (in case the distillation thermometer is one calibrated for total immersion) is suspended so that the bulb is adjacent to and located at the center of the exposed portion of the distillation thermometer stem. Finally, cold water is placed in the condenser (in case a trough condenser is used) or is circulated through the condenser (in case a closed-jacket condenser is employed). A small flame now is lighted beneath the flask and the oil is brought carefully to the boiling-point, so that the distillation starts at the proper rate.

The temperature of the vapor at the side-arm of the flask when the first drop falls from the outlet end of the condenser is recorded as the *Over-Point.*

During the distillation, the flame is regulated so that the distillation proceeds at the rate of 2.5 c.c. per minute until a temperature of 300°C. (572°F.) is reached.

In the course of the distillation the following observations are made:

1. The auxiliary temperatures are read for each rise of 50°C. (90°F.).

2. At successive 25°C. (45°F.) intervals the volume of the distillate is read. For example, if the over-point of an oil is 60°C. (140°F.), the next reading is taken at 75°C., then at 100°C. (212°F.), and thereafter at 25° intervals as indicated.

3. When a temperature of 100°C. (212°F.) is reached (provided, of course, the over-point is below this temperature), the receiver at the outlet of the condenser is removed and replaced by another. This procedure is repeated when each of the temperatures 150°C. (302°F.), 200°C. (392°F.), 250°C. (482°F.), and 300°C. (572°F.) is reached. After the distillation has been completed, these fractions are dealt with as follows: The five (or less, depending upon the nature of the crude oil) fractions are first tested for Baumé gravity and for index of refraction. Next, the fractions collected from the over-point to the 150°C. (302°F.) point are mixed together, and the Baumé gravity, index of refraction, and loss to sulphuric acid are determined. The "doctor test" also is made. Finally, the fractions ranging from

150°C. to 300°C. (572°F.) are mixed together, and the volume, Baumé gravity, index of refraction, and loss to sulphuric acid are determined, and the "doctor test" is applied as before.

When the temperature of 300°C. (572°F.) is reached, the distillation is stopped temporarily. The flask now contains the residue boiling above 300°C., in which are present lubricating oils, and, perhaps, asphaltene and paraffin wax.

After cooling slightly, the flask is weighed, a 10 c.c. sample is poured out, and the flask is weighed again. This sample is used in the estimation of asphaltene (see page 19). The flask then is stoppered; the side-tube is attached to an air condenser, and the distillation is continued rapidly to dryness. The distillate collected is used in the estimation of paraffin wax (see page 18).

After cooling, the flask is weighed. In calculating the percentage of coke left in the flask, the 10 c.c. sample removed for the purpose of estimating asphaltene should be taken into account.

Notes and Precautions.—In order to secure concordant results by this method, the directions should be observed strictly, draughts eliminated, and the rate of distillation held constant. The receivers are preferably graduated to 0.1 c.c. and the volume of the fractions should be measured at a temperature of 60°F.[1]

The accuracy of the thermometers should be known. Thermometers which are in continuous use may be calibrated by the U. S. Bureau of Standards method.[2] Thermometers are manufactured which are calibrated for the depth of immersion found in the Engler flask, and for such thermometers no auxiliary temperatures need be taken, the subsequent calculation of corrections to the observed readings being eliminated.[3] A reading of the barometer, taken just before or immediately after the distillation, also should be included in the data, and, if necessary, the observed temperature may be corrected to normal pressure by means of the equation given on page 52.

[1] In conducting distillation tests on heavy viscous crude petroleums it is not practicable to measure the volume of the charge; but it is sufficient merely to weigh into the flask a quantity which amounts to approximately 100 c.c. The volume of the crude oil sample then may be calculated from the known specific gravity of the petroleum.

[2] Bureau of Standards *Circular* **8**, 1911.

[3] Concerning corrections to observed readings of thermometers calibrated at total immersion, see page 52.

The Methods of the United States Bureau of Mines.—Rittman and Dean[1] have conducted researches on the analytical distillation of petroleum, comparing methods of minimum condensation (Allen-Jacobs method), moderate condensation (Engler-Ubbelohde method), and maximum condensation (Rittman-Dean method). As a result of comparison, the fractionation efficiency

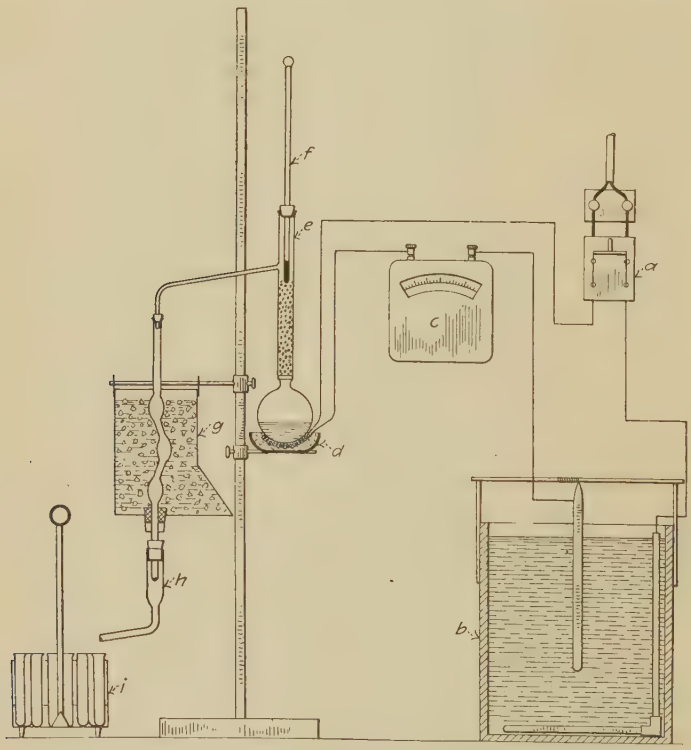

FIG. 5.—Diagram of Rittman-Dean distillation apparatus assembled, with electric heater and water rheostat.

a, switch at electric mains; *b*, water rheostat; *c*, ammeter; *d*, heater; *e*, distilling flask, with Hempel column; *f*, thermometer; *g*, condenser, with three-bulb tube and ice-filled jacket; *h*, adapter; *i*, metal rack containing receiving tubes packed in ice. An ordinary Liebig condenser may be used and the beads in the fractionating column may be replaced by magnesium or aluminum turnings or by several cones of wire gauze.

of the Rittman-Dean method was placed at 56 per cent., the Engler-Ubbelohde at 26 per cent., and the Allen-Jacobs at 14 per cent.

In the apparatus of Rittman and Dean a Hempel fractionating

[1] Bureau of Mines *Bulletin* **125**.

column is interposed between the flask and the condenser.[1] For this purpose the standard Hempel creosote flask may be used.

The *apparatus of Rittman and Dean* is shown in Fig. 5. This includes an electric heater, instead of which, of course, a suitably shielded gas burner may be used. Preparation for the distillation is made by counterpoising on a balance the flask, a spiral of iron gauze, and the beads to be used in the column. The charge of oil then is weighed in and the fractionating column is adjusted.

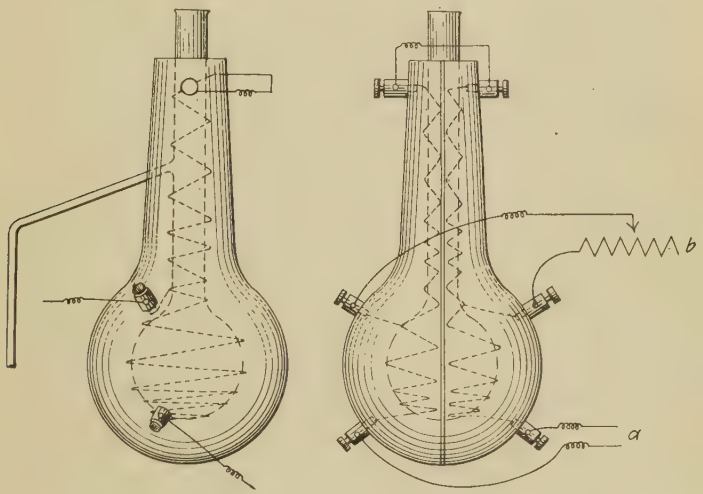

FIG. 6.—The Allen and Jacobs apparatus, modified to permit independent control of the quantities of heat supplied to the bulb and neck of the distilling flask.

a, Wires connecting with the electric mains; *b*, variable resistance shunted across the top of the bulb.

The Allen-Jacobs method[2] was evolved primarily for the distillation of heavy crude oils, such as those of California, which contain large quantities of lubricating oils, asphalt, and often water. The novelty of this apparatus consists in an insulated, electrically heated flask shown in Fig. 6. The complete installation is illustrated in Fig. 7. The procedure of operation

[1] It is of interest to note here that some petroleum chemists carry out distillations by means of the Engler-type flask, placing a column composed of pieces of porous plate or of glass beads in the neck of the flask below the outlet tube.

[2] Bureau of Mines *Bulletin* **19.**

is as follows:[1] The oil is distilled under atmospheric pressure at increasing temperatures up to 325°C. (617°F.), the receivers being changed at each increment of 25°C. (45°F.) The temperature then is permitted to drop to 125°C. (257°F.), to keep the oil from boiling over when the vacuum is used, and the distillation again is continued under a vacuum of 10 to 20 mm. mercury pressure, until the temperature within the flask reaches 325°C. (617°F.). The fractions boiling to 150°C. (302°F.) under atmospheric pressure comprise the *unrefined*

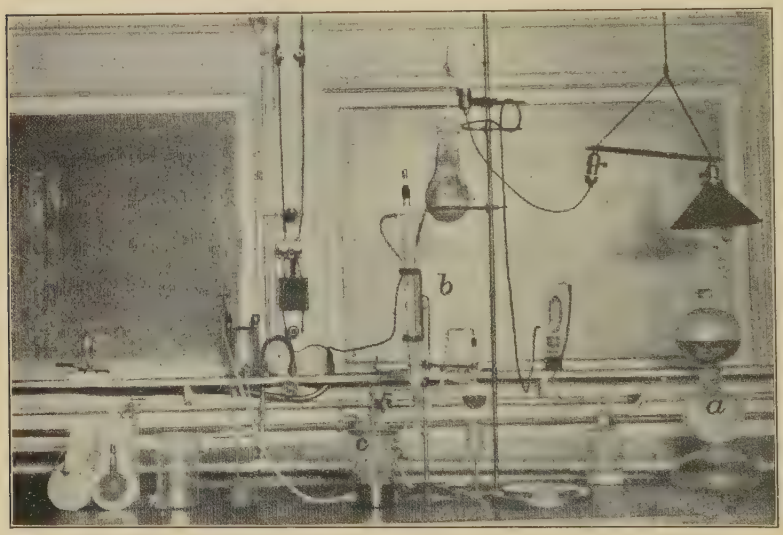

FIG. 7.—The Allen and Jacobs apparatus (complete), showing the electric still and accessories.

a, Apparatus for generating carbon dioxide; *b*, Liebig condenser with water-heater; and *c*, Brühl receiver.

naphthas. Those boiling between 150°C. (302°F.) and 300°C. (572°F.) under atmospheric pressure constitute the *unrefined lamp oils*. Those fractions boiling between 300°C. (572°F.) under atmospheric pressure and 325°C. (617°F.) under a vacuum of 10 to 20 mm. mercury pressure, are termed the *unrefined lubricants*. The portion remaining in the flask is referred to as *asphaltum residue*.

12. Estimation of Paraffin Wax.—The fraction collected for the determination of paraffin wax in the above distillation is examined as follows by Holde's method.

[1]Bureau of Mines *Tech. Paper* **74**. If desired, the apparatus of Rittman and Dean may be employed in the procedure of Allen and Jacobs.

Apparatus and Reagents.—Large test tube; reservoir for salt and ice mixture; solvent consisting of equal parts by volume of absolute ethyl alcohol and anhydrous ethyl ether; apparatus of Fig. 8, which consists of a funnel so arranged in a bath that it may be surrounded by salt and ice mixture while filtration is going on; filter papers; thermometer reading to −20°C. (−4°F.); Erlenmeyer flask; Liebig condenser; benzene; and analytical balance.

Procedure.—Three to five g. of the sample are weighed into the test tube and dissolved in the absolute alcohol ether mixture sufficient of the latter being added to give a clear solution. The solution then is cooled in salt and ice mixture to a temperature of −20°C. (−4°F.), and sufficient of the absolute alcohol-ether mixture (cooled also to −20°C.) is added to bring all oily drops into solution, leaving only flakes of paraffin visible. After cooling for 30 minutes, the precipitated paraffin is filtered with slight suction on the funnel surrounded by a freezing-mixture of salt and ice. The precipitate is washed well with cooled absolute alcohol-ether solution[1] and then dissolved in hot benzene, which is run into a small Erlenmeyer flask. The solvent (benzene) is distilled off through a condenser, and the flask and its contents are heated to 100°C. (212°F.) for 30 minutes in an oven. The flask and the wax which it contains are cooled in a desiccator, and then are weighed. The percentage by weight of wax in the residue above 300°C. (572°F.) is calculated, and then the percentage by weight of paraffin wax in the original crude petroleum is found.

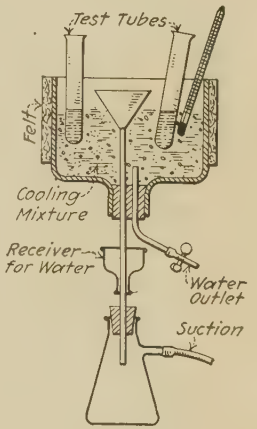

FIG. 8.—Paraffin filtration funnel.

13. Estimation of Asphaltene.[2]—The sample set aside (during the crude distillation) for the purpose of estimating asphaltene, is examined as follows.

Apparatus and Reagents.—500-c.c. glass-stoppered bottle;

[1] The precipitate on the filter is washed until free from oil and should be stirred occasionally during the washing operation.

[2] See also under *Asphalt*, page 142. The method described there is to be preferred because of its accuracy.

close-texture quantitative filter papers; Erlenmeyer flask; Liebig condenser; funnel; analytical balance; benzene, and spirit prepared from natural gas gasoline.

Procedure.—Five to ten g. of the sample are weighed into the 500-c.c. bottle and shaken with 40 volumes of spirit prepared from natural gas gasoline and having a boiling-point between 40°C. (104°F.) and 95°C. (203°F.). The mixture is permitted to stand in a cool, dark place for twenty-four hours, when it is decanted through a double layer of filter paper and washed well with the spirit.

The asphaltene on the filter is dissolved in hot benzene, the resulting solution being run into a small Erlenmeyer flask; most of the solvent is distilled off through a condenser, and the flask and its contents are heated in an oven at 100°C. for 30 minutes. After the flask and the residue have been cooled in a desiccator, they are weighed. The percentage of asphaltene in the residue above 300°C. (572°F.) is calculated, and from this result the percentage of asphaltene in the original crude is ascertained.

14. Other Tests Concerning Crude Petroleum.—The *formolite number* of crude oils and their fractions sometimes is determined, and is specific for straight-chain olefines which give a brown syrupy liquid and for closed-chain olefines and for the aromatics which yield solid polymerization products ("formolite"). By the method of Marcusson[1], 27 g. of the oil are dissolved in 50 c.c. of petroleum spirit (previously treated with sulphuric acid and formalin), 30 c.c. of concentrated sulphuric acid are added, and the mixture is cooled in ice water. Fifteen c.c. of formalin then are added and the mixture is shaken with cooling until heat ceases to be evolved, after which it is added to 200 c.c. of ice water. The whole next is neutralized with ammonia in excess; and finally the precipitate is collected on a Büchner funnel, washed first with naphtha, then with water, dried at 105°C. (221°F.), cooled and weighed. The formolite number is the number of grams of dried precipitate from 100 c.c. of the oil. Clifford Richardson[2] has developed a modified method for the formolite reaction, whereby the quantity of unattacked oil is determined.

The *iodine number* and the *loss to sulphuric acid* are two deter-

[1] *Chem.-Ztg.*, **35** (1911), 729 and 742. See also Herr, *ibid.*, **34**, 893.
[2] *J. Ind. Eng. Chem.*, **8** (1916), 319.

minations having to do mainly with the unsaturated hydrocarbons in the fraction under examination. The determination of the loss to sulphuric acid is discussed on page 57. For the determination of the iodine number, the method of Wijs[1] is reliable, although other methods may be used with good results.[2] The iodine number does not represent the actual percentage of unsaturated compounds present: a high iodine number indicates either the presence of a considerable amount of compounds having one double-bond or a smaller proportion of more highly unsaturated compounds.

The determination of the *composition of petroleum* from various sources generally resolves itself into a process of fractional distillation. Considerable knowledge has been gained with respect to the lower fractions of petroleum, naphthene hydrocarbons (cyclo-pentane, cyclo-hexane and their derivatives), paraffins, aromatics and olefines having been isolated; while the presence of cyclo-olefines and acetylenes has been indicated.[3] As to the pressed lubricating fractions, it is thought that they are composed of naphthenes, polynaphthenes, and polymerized olefines.[4]

Olefines are polymerized to heavy liquids by the formolite reagent.[5] According to Engler,[6] the olefines may be removed by

[1] *Ber.*, **31** (1898), 750. The Wijs iodine solution is suitable for the determination of the iodine absorption of all hydrocarbon oils and the test is carried out exactly as for fatty oils, but it is often necessary to use a measured excess of solution and to make a blank test side by side with a known specimen, to eliminate the time factor, since in many cases definite absorption values are not obtainable, but the figure is largely proportional to the time of action. For particulars regarding the Wijs method, see LEWKOWITSCH'S "Chemical Technology and Analysis of Oils, Fats and Waxes," 5th ed., **1,** 392–416; FRYER and WESTON'S "Technical Handbook of Oils, Fats and Waxes," **2,** 92-6; HOLDE-MUELLER'S "Examination of Hydrocarbon Oils," **1915,** 352; ALLEN'S "Commercial Organic Analysis," 4th ed., **2,** 31; and LUNGE-KEANE'S "Technical Methods of Chemical Analysis," **3,** i, 116–19. See also, in this connection, RADCLIFFE and POLYCHRONIS, *J. Soc. Chem. Ind.*, **35** (1916), 340; and *J. Ind. Eng. Chem.*, **11** (1919), 1164.

[2] See SHERMAN'S "Methods of Organic Analysis," **1905,** 130 *et seq.;* and DEAN, Bureau of Mines *Tech. Paper* **181.** See also BOHRISCH and KÜRSCHNER, *Apoth.-Ztg.*, **33** (1918), 247, 251, 257, 262, 266, and 272.

[3] On the determination of *hydrocarbon groups,* see BACON and HAMOR, *lib. cit.*, **2,** 521.

[4] DUNSTAN and THOLE, *J. Inst. Pet. Tech.*, **1918,** 191; and BROOKS and HUMPHREY, *J. Am. Chem. Soc.*, **40** (1918), 822.

[5] GURWITSCH'S "Wissenschaftliche Grundlagen der Erdölbearbeitung," 47.

[6] *Proc. Int. Pet. Congress,* **1912.**

shaking with a concentrated mercuric acetate solution. Liquid sulphur dioxide, employed in accordance with the method of Edeleanu, is an approximately selective solvent for olefines, aromatics, sulphur compounds and other impurities, leaving naphthenes and paraffins.[1]

The aromatic hydrocarbons may be removed from the gasoline fraction, and the dinitrobenzene and dinitrotoluene recovered by the following method: The fraction is treated with a mixture of one part of fuming nitric acid and three parts of concentrated sulphuric acid at a temperature below 60°C. (140°F.).[2] The reaction mixture is poured on ice, and the precipitated nitro-compounds are recrystallized from ethyl alcohol. Aromatic hydrocarbons, according to Engler, also may be removed by treating the distillate with triphenyl-methane or picric acid. Fuming sulphuric acid acts upon the aromatic hydrocarbons, and slowly upon the paraffins at room temperature.

The paraffins and naphthenes are, in fact, but slowly acted on by all reagents, at ordinary temperatures, with the exception of chlorine and bromine. Having removed the hydrocarbons of other series, the paraffins and naphthenes, as well as the individual members of both series, are separated by fractional distillation and the fractions are identified by boiling-point, specific gravity, and index of refraction determinations. For examples of researches along this line, see the reports of Mabery,[3] Markovnikov,[4] and Gurwitsch.[5] The theory of distillation in detail may be found in Young's "Fractional Distillation," and in the reports of the later researches of Rosanoff.[6] For an example of the fractional distillation of cracked gasoline by I. W. Humphrey, see the *Appendix*, page 334.

II. THE COMMERCIAL EVALUATION OF CRUDE PETROLEUM

The commercial evaluation of crude petroleum involves primarily the determination of the quantity and quality of merchantable products obtainable from it. Knowing this, the

[1] See Bacon and Hamor, *lib. cit.*, **2**, 594; and Tausz and Stüber, *Z. angew. Chem.*, **32** (1919), I, 175. See also pages 57 and 62.

[2] *Ann.*, **301**, 157.

[3] *Am. Chem. J.*, **33** (1905), 251.

[4] *Ann.*, **301**, 157.

[5] *Lib. cit.*, **1913**, parts i and ii.

[6] *J. Frank. Inst.*, **172** (1911), 527–44.

cost of a barrel of crude oil delivered to the refinery is compared with the sum of the prices which may be obtained for—that is, the market value of—the products secured from this same barrel of oil, less the cost of their production.[1]

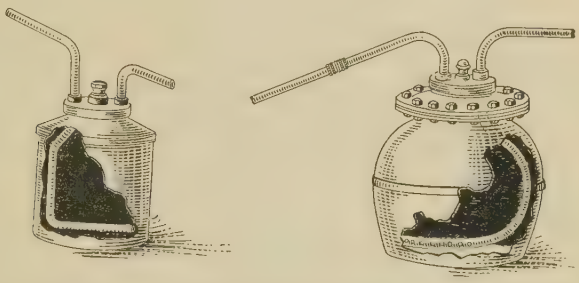

FIG. 9.—Oil stills for laboratory work, showing the patterns of ½-gal. (left) and large (right) sizes. It is desirable that laboratory stills of these types be made with steel or iron bottoms, particularly when used for distillations *in toto* to coke.

1. Preliminary Examination.—In order to simplify the procedure involved in the final examination, a preliminary examination of the crude petroleum is made by the aid of certain of the methods previously described in this Chapter. Final reports should be presented in neat tabular form, with the distillation curve attached to the data sheet.

The data secured by the preliminary examination may advantageously include the following:

(*a*) Source of sample. If an oil-well, state the depth of sand.

(*b*) Specific gravity of the crude petroleum, together with Baumé scale equivalent, at 60°F.

(*c*) Color, "bloom" and odor.

(*d*) Viscosity.

(*e*) Percentage of water and B.S.

(*f*) Percentage of sulphur.

(*g*) Distillation analysis: (1) Over-point. (2) Percentage by volume, index of refraction, and Baumé gravity of the initial fractions as follows (uncorrected temperatures): over-point to 100°C. (212°F.), 100°C. to 150°C. (302° F.), 150°C. to 200°C. (392°F.), 200°C. to 250°C. (482°F.), and 250°C. to 300°C. (572°F.). (3) Percentage by volume, loss to sulphuric acid (see page 57), index of refraction, and Baumé gravity of the blended fractions as follows (uncorrected temperatures): over-

[1] See BACON and HAMOR, *lib. cit.*, **2**, 497–501.

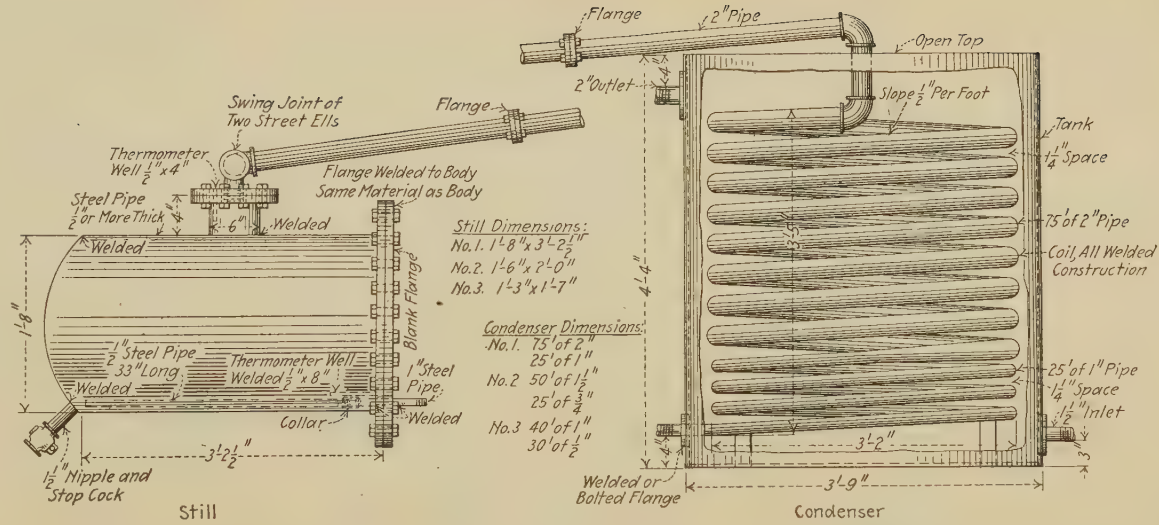

FIG. 10.—Stills and condensers.

The stills are of all welded construction from 15″, 18″ and 20″ steel pipe, respectively. In addition to the arrangements shown, it will be found to be advantageous to have a pet-cock located on the front of the still, near the bottom, for the purpose of removing samples as the distillation proceeds. The ½″ pipe shown in the bottom of the still is the steam-line, which should be perforated with ⅛″ holes, 1″ apart, along the length within the still. If preferred, this line may have its inlet at the top of the still, the vertical section terminating in two lateral sections of perforated pipe at the bottom. When the still is used with air for treating residues to produce asphalt, the steam-line at the bottom of the still may be used for introducing the air, but, in addition, there should be a steam-line (top-steam) above the surface of the oil, for injecting steam into the vapors and thus preventing explosions. A safety-valve also should be placed on the vapor-line from the still.

point to 150°C. (302°F.) and 150°C. to 300°C. (572°F.). (4) Distillation curve, plotting temperatures (corrected; see page 52) against corresponding percentages, from the over-point to the 300°C. (572°F.) point.

(*h*) Percentage by weight of the residue boiling at 300°C. (572°F.) and above.

(*i*) Percentage by weight of asphaltene.

(*j*) Percentage by weight of paraffin wax in the residue above 300°C. (572°F.) and equivalent percentage in the original crude oil.

(*k*) Percentage of coke.

(*l*) Percentage of nitrogen.

2. Final Examination.—In order to separate and refine the commercial products existing in the crude, it is considered good practice to treat samples of one barrel in amount, although, of

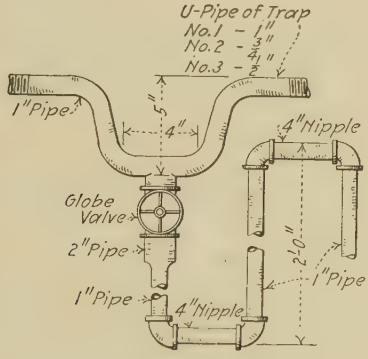

FIG. 11.—Water-traps to be attached to the outlet of the condenser during steam distillation.

course, smaller samples may be used where the larger scale apparatus or sufficient oil is not available. For the treatment of crude oil in one barrel lots, apparatus similar to that shown in Figs. 10 to 15, inclusive, may be used.

A method which often is employed is first to distil the oil and separate into 2 per cent. fractions, which then are blended successively to give the various products. With the data from the preliminary examination at hand, and especially when treating samples as large as one barrel in size, the procedure of cutting the stream from the condenser into 2 per cent. fractions may be dispensed with. There follow several methods of treat-

ment based upon the data secured in the preliminary examination.[1]

(*A*) Straight Paraffin-Base Crude Oil: *Method* 1.—Distillation by means of direct bottom-steam[2] and heat applied to the bottom of the still. When the gravity of the stream from the condenser reaches 54°Bé. (temperature in still about 475°F.), the receiver is changed. The distillate secured is termed *crude naphtha* and is fractionated into *gasoline* of the proper end-point and (optionally) *naphtha distillate* of boiling-point 150°C. to 240°C. (464°F.). The bottoms remaining in the still after the separation of the naphtha distillate are mixed with the *natural lamp oil distillate*. Finally, the gasoline and naphtha distillate fractions are refined with sulphuric acid by the methods described on page 34. The finished products are measured and tested by the methods given in the following chapter, to define their quality.

When the gravity of the stream from the condenser arrives at 41°Bé., the receiver is changed again. The product is termed *natural lamp oil distillate* (burning oil distillate or kerosene distillate) and is refined with sulphuric acid by the methods de-

[1] Consult Bacon and Hamor, *lib. cit.*, **2**, Chaps. 12 and 13. See also page 13.

[2] The amount of steam to be used in distillations is a variable factor. Different amounts are necessary for different crude petroleums under examination, depending upon their physical properties and the qualities of the products desired.

In running a crude oil, using fire and steam, it is customary not to use any steam in the still until the temperature has reached approximately 300°F. At this point, only a very small amount of steam is admitted and the water condensed will probably amount to only 1 per cent. of the total condensate. The amount of water condensed when the end of the gasoline fraction is reached will be approximately 5 per cent. or an average of about 3 per cent.

At the conclusion of the kerosene cut, about 10 per cent. of steam should be used, increasing to 20 per cent. at the end of the wax distillate cut. In running the wax distillate too large a volume of steam should not be used, since this would result in the distillate containing a large amount of amorphous wax which would interfere with good pressing and sweating. In running the bottoms of the still to cylinder stock it is necessary to use a large amount of steam. In the case of cylinder stock, which is usually run to a certain flash-point, 500°F., for example, the temperature of the still is held at about 30°F. above that of the resulting flash-point, which, in this case, would be 530°F. By running in this manner, a large amount of the condensate will be water, especially at the end of the run, where it may be as high as 95 per cent., only about 5 per cent. of the condensate being oil.

scribed on page 34. If the flash-point is too low, the product may be steamed to the proper test. The finished product is measured and tested by the quality-defining methods given in the chapter on illuminating oils (see page 68).

When the stream from the condenser reaches 38°Bé., the receiver is changed another time. The distillate is termed *mineral seal stock*, which is rerun to make *mineral seal distillate* and this latter is refined with sulphuric acid. The finished product is measured and tested by such methods as will define its quality (see page 34).

When the stream comes to a gravity of 36.5°Bé., the receiver is changed once more and the distillate is termed *gas and fuel oil stock*.

When the stream attains 33°Bé., the distillation is stopped.[1] The fraction is designated *lubricating distillate* and the residue *cylinder stock*.

The *cylinder stock* is filtered through Florida earth and the product, *cylinder oil*, is tested for viscosity, flash-point and color, and the asphaltene content is determined.

The *lubricating distillate* is cold-pressed once, or perhaps twice, at a temperature of 5° to 20°F., to remove the *slack wax*.[2] The filtrate is termed

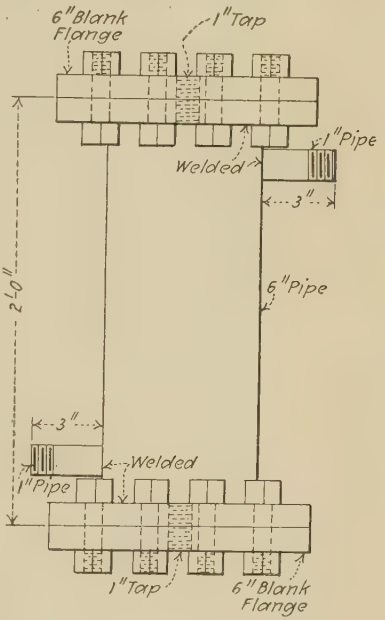

FIG. 12.—Fractionating segment. Segments of this design may be fabricated into almost any form of fractionating column.

[1] In the ultimate refinery operations the distillation will be continued until the "bottoms" have reached a desired flash-point—500° to 600°F.

[2] If the lubricating distillate is cooled slowly before the pressing, larger crystals of paraffin will form and the pressing operation may be carried out with greater ease and effectiveness. The oil is cooled by first using ice and later a salt and ice mixture. While the pressing is being done, the press is placed in a box on the bottom of which is some ice, to prevent too rapid rise in temperature during the pressing operation. A letter-press, tincture press or small industrial filter-press having a capacity about equal to the amount of wax in the distillate, may be used. The lubricating distillate may be "cracked" by distilling rapidly with dry heat, in order to secure an oil that will "press" well.

pressed lubricating distillate and is treated by first redistilling with steam and separating into 10 per cent. fractions until 70 to 80 per cent. of the oil has been distilled, when the operation is discontinued. The flash-point, viscosity and color of the fractions (including the residue) are deter-

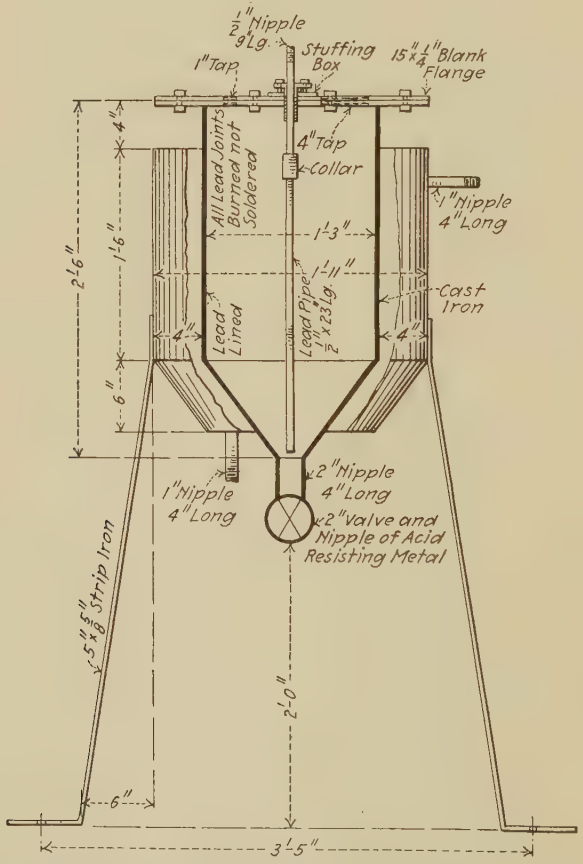

FIG. 13.—Water-jacketed agitator.

By means of this apparatus the temperature during refining may be controlled, and the agitation may be done by means of either an air-blast or a mechanical stirring device. When agitating by means of an air-blast, the lead pipe shown is perforated at its lower end.

mined. Accepting the viscosity of the residue as the standard for *high viscosity lubricating distillate* from the particular crude oil, the distillates then are blended to give *low viscosity lubricating distillate, medium viscosity lubricating distillate,* and possibly *gas*

and fuel oil stock from the lowest fractions, depending upon their color, flash-point and viscosity. Finally, the blended lubricating distillates are divided each into two portions, one of which is refined by means of Florida earth only, and the other by means of sulphuric acid followed by Florida earth. The finished products are tested by such methods as will define their quality.

The *slack wax* secured by cold-pressing the *lubricating distillate,* is sweated fractionally in an oven, increasing the temperature at the rate of 2°F. per hour, until the melting-point of the residue reaches 126° to 128°F. This sweating process is carried out in

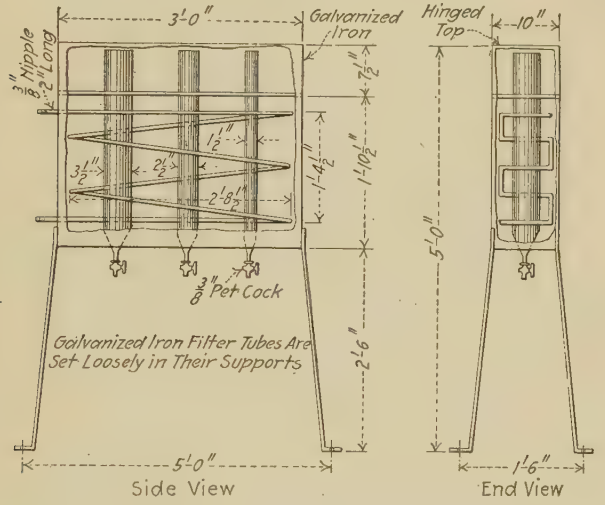

FIG. 14.—Filter tubes and oven. The oven may be heated by means of the steam-coil.

pans such as those shown in Fig. 15. Hot water is introduced into the pans until the surface is just above the screen. The melted wax then is poured in and the whole is allowed to cool slowly until the wax has solidified, when the water is withdrawn. The sweating then is begun.

As the sweating proceeds, the stream is cut into 5 per cent. fractions and the melting-points are determined. The lower fractions are termed *sweat oil* ("foots oil"), while the higher ones are blended to produce a wax of 118° to 120°F. melting-point.[1] The crude waxes finally are filtered through Florida earth to give

[1] Concerning the sweating of wax, see the *Appendix,* page 493. On the color of paraffin wax, see page 175.

the finished products, which are examined for color.[1] Bauxite (1 lb. of ignited mineral per 1 gal. of melted water-free wax) is said to be an efficient decolorizing medium for wax as well as for lubricants.

Method 2.—This is essentially the same as that described under *mixed-base crude, method* 1, *infra.*

(*B*) Mixed-Base Crude Oil: It should be borne in mind that, as the nature of crude petroleum varies from the straight paraffin-base type through the mixed-base to the asphaltic, the fractions become heavier for the same range of boiling-point.

Method 1. *Dry Distillation.*—The cuts are made on a temperature basis, instead of on that of Baumé gravity, as follows: The first cut is made when the temperature of the oil in the still reaches 475°F. (246°C.). This fraction represents the *crude*

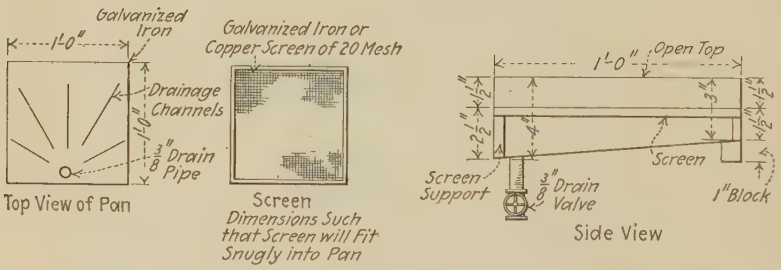

Fig. 15.—Sweating pan.

naphtha, and it is fractionated into *gasoline* and *naphtha distillate.* Any residue from the last-mentioned distillation is mixed with the *natural lamp oil distillate.* The *gasoline* and *naphtha distillate* are refined with sulphuric acid in the usual way, measured, and tested for quality.

The next cut is made when the temperature in the still reaches 625°F. (329°C.). The distillate secured is termed the *natural lamp oil distillate*, and is refined with sulphuric acid in the customary manner, measured, and then examined for quality.

The distillation now is continued slowly ("cracking") until the temperature in the still reaches 700°F. (371°C.), when the operation is discontinued. The fraction collected is termed *cracked distillate*, and is fractionated into *crude naphtha, standard*

[1] In some cases it will be necessary to resweat the scale to attain the best color results, or to treat the sweated paraffin with sulphuric acid before the final decolorizing filtration through charcoal, fullers' earth or bauxite.

white distillate, and *gas and fuel oil stock*. The first two products are refined with sulphuric acid in the usual way, redistilled with steam, treated again with sulphuric acid or with sodium plumbite, measured, and tested for quality. The *gas and fuel oil stock* merely is measured.

The residue in the still (*tar*) is distilled rapidly to dryness in a smaller still. The fractions are known as *cracked distillate* and *paraffin distillate*. The last heavy runnings from the still are termed *wax tailings* and merely are weighed. The coke remaining in the still is removed and weighed.[1]

The *paraffin distillate*, secured as directed in the last paragraph, is cold-pressed at a temperature of 15°F., redistilled with steam, and cut into fractions and further treated as described under *paraffin-base crude, method* 1 (page 28).

Method 2. *Direct bottom-steam utilized during the distillation.*—The general procedure is practically the same as that of *method* 1, *paraffin-base oil*, with the following exceptions: The cuts are made by temperature instead of by Baumé gravity; the *crude naphtha* fraction is cut when the temperature in the still is 330°F. (166°C.); *natural lamp oil distillate* is cut when the temperature in the still reaches 500°F. (260°C.); and *lubricating distillate* is cut when the temperature in the still[2] comes to 620°F. (327°C.). The residue remaining in the still is unsatisfactory *per se* as a *cylinder oil stock*, and is refined further or is either utilized as a road-binder or treated by air-blowing to produce a harder product.

Method 3. *Skimming Process.*—The procedure is essentially the same as *method 1, mixed-base crude oil*, except that distillation is discontinued when a temperature of 700°F. (371°C.) is attained in the still. The distillates are treated as described, but the residue in the still is considered as belonging with the fuel oil, and merely is measured with the *gas and fuel oil stock* secured from the *cracked distillate*.

[1] On petroleum coke, see Bacon and Hamor, *lib. cit.*, **2**, 859. The examination of petroleum coke should include the determination of specific gravity, porosity (Langbein, *Chem.-Ztg.*, **1906**, 1115), calorific value, and proximate composition (as for coal: *J. Ind. Eng. Chem.*, **9** (1917), 100). The determination of iron content also is of importance. The loss on extraction with carbon disulphide sometimes is found.

[2] In the ultimate refinery operations the distillation will be continued until a certain gravity of the "bottoms" has been reached. This will be gauged by the Baumé gravity of the stream or of the "bottoms."

(*C*) Asphalt-Base Crude Oil[1]: Asphaltic crudes may be treated by (1) *Method 3, mixed-base crude oil,* or by (2) *Method 2, mixed-base crude oil.*

(*D*) Pressure Distillation: Preliminary distillations are made on the crude oil at atmospheric pressure, for the purpose of securing sufficient residue boiling above 300°C. (572°F.) to make up the volume necessary for the pressure distillation.[2]

Procedure.—Seven liters of the residue above 300°C. are weighed into the still (see Fig. 16), the cover and gasket (copper or asbestos) are bolted on, the valve at the outlet of the condenser is closed, and the burner is lighted under the still. When the

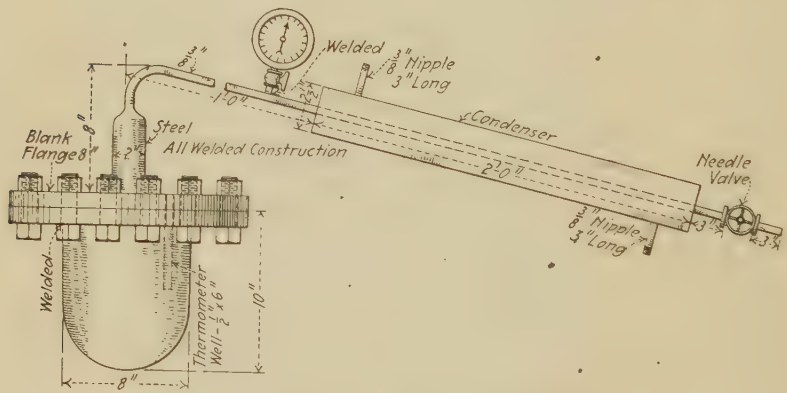

Fig. 16.—"Cracking" still for experimental use.

pressure on the gage reaches that at which it is desired to conduct the distillation, the valve is opened slightly and the flame is so regulated that the entire distillation is carried out at the desired pressure. The distillation is continued[3] until ⅔ of the charge has been collected as distillate. After cooling, the cover is removed and the body of the still is weighed. In order to secure complete data, a series of distillations should be carried out on the residue from the crude, at 50, 75, 100, 125 and 150 pounds to the square inch above atmospheric pressure.

[1] The yield of asphalt from asphaltic petroleum may be determined by the use of the Brown evaporator. The controlling factor is oil heated to a temperature of 565°F., which temperature can be maintained more effectively than by means of an air-heated asphalt oven. *Cf.* page 136.

[2] In some cases it may be desirable to conduct the pressure distillation test on the gas oil fraction instead of on the residue above 300°C.

[3] At the rate of from 15 to 18 per cent. per hour.

The distillate secured during pressure distillation is redistilled at atmospheric pressure until the temperature of the vapor has reached 225°C. (437°F.). The distillate consists of the gasoline produced by cracking and may have a high refining loss, disagreeable odor and a yellow color, the two latter characteristics being wholly or partially eliminated during the refining. The residue will consist of gas and fuel oil stock, and, in the case of the pressure distillation, the residue may contain considerable coke.

The crude gasoline secured by pressure distillation is refined with 3 to 5 pounds of concentrated sulphuric acid per barrel of

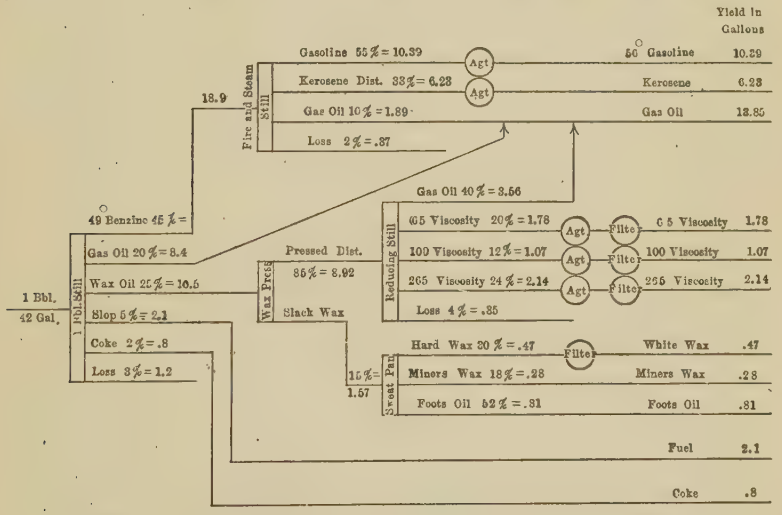

FIG. 17.—Flow-sheet of one-barrel treatment of representative Mid-Continent crude petroleum. (*Courtesy of P. M. Miskell and J. W. Coast.*)

product, by the method described under *mixed-base crude oil, method* 1, page 31, followed by redistillation and then treatment with sodium plumbite. The refined product is measured and examined for quality.

The percentage of coke formed during the pressure distillation is determined by diluting the residue with gasoline, filtering, washing the residue with carbon disulphide, drying in an oven, and finally weighing.

Reports of results should include a curve showing the variation of cracked gasoline yield with variation of pressure.

3. Notes and Practical Suggestions.—*Quality of Products Obtainable from Various Crude Oils.*—From the straight paraffin-

base crude petroleums high-grade illuminating oils, low and medium viscosity lubricating oils, and steam cylinder oils are obtained. The mixed-base crude oils vary in the grades of products which are obtainable, but products of high quality are secured therefrom. The Texas and Louisiana coastal crude petroleums furnish high-grade lubricating oils of low cold test and high viscosity. Much asphalt, road oil and fuel oil also are produced from the crude oils of Texas and California.

Treatment of Crude Products.—The sulphuric acid used in the refining of the crude products obtained by the foregoing methods should possess a specific gravity of 1.84 (66°Bé.); it should be free from nitrogen oxides and selenium. The quantity of acid, method of treatment and time factors involved will necessarily vary with different crude oils as well as with the methods of distillation. Cracked distillates may be satisfactorily refined by a treatment with sulphuric acid, followed by distillation with steam and a final treatment with sulphuric acid. Even with this treatment it is difficult, however, to secure a product which is comparable in quality with the refined natural distillate, especially as regards color. Lamp oil or kerosene fractions which contain aromatic compounds will demand special treatment, such as the use of fuming sulphuric acid or of liquid sulphur dioxide,[1] in order to remove the "smoky constituents." It is also possible that fractions obtained from crude petroleums containing oxygen compounds may be treated advantageously with strong alkali before the agitation with sulphuric acid. Depending upon the method of distillation employed and the nature of the original crude, finished lubricating oils sometimes may be secured by simply filtering through Florida earth, but in other cases the filtration must be preceded by treatment with sulphuric acid. *Gasolines* and *naphtha distillates* may be refined by using from 2 *to* 5 *pounds* of sulphuric acid to the barrel of product treated; *natural lamp oil distillates* by the use of from 3 *to* 10 *pounds* of sulphuric acid to the barrel of oil treated; and *crude lubricating oils* by using 10 *to* 40 *pounds* of sulphuric acid to the barrel of product treated.

[1] A method which is not used in the American petroleum industry. For a description of this process, see Bacon and Hamor, *lib. cit.*, **2**, 593. On its use in the laboratory, see Joseph, *Trans. Utah Acad. Sci.*, **1**, 214. On the use of the Edeleanu method of separation in the case of motor gasoline, see page 62.

Refining with sulphuric acid includes the treatment with sulphuric acid and the subsequent washing with water and alkali. The crude product is placed in an agitator, which may be of the design shown in Fig. 13. A small portion of the total amount of acid to be used is added and the agitation is carried out by means of an air-blast. The acid then is permitted to settle and the sludge is drawn off. The remainder of the acid is added and the agitation is continued for the proper length of time. Settling is allowed until only traces of suspended matter are apparent in a sample taken from the upper layer in the agitator. The acid sludge then is drawn off and the oil is washed by spraying with large quantities of water, followed by agitation with water. Finally, the product is agitated with from 1 to 2 per cent. of sodium hydroxide solution of a gravity of 6° to 14°Bé. (4 to 9.5 per cent.). After settling, the sludge is withdrawn and the oil is washed with water by agitation.

The temperature at which the agitation with acid is carried out should not be so high that excessive oxidation takes place, nor so low that settling of the acid is prevented. The temperature will vary (preferably) between 40° and 90°F., depending upon the character of the distillate treated. In the refining of crude lubricating oils by means of sulphuric acid, difficulties may be encountered on account of the formation of troublesome emulsions after the alkali treatment. In order to minimize the likelihood of the formation of emulsions, the alkali solution and the body of the oil are mixed hot, and the final washing is accomplished by means of hot water. It is also recommended for lubricating oils that a stirring apparatus be substituted for the air-blast.[1] Emulsions often may be broken up by means of heat supplied by a closed steam coil.

The time factors in refining will necessarily vary according to the density of the distillate under treatment, the method of distillation and the nature of the original crude oil. In the refineries of northwestern Pennsylvania time factors for illuminating oils have been given as follows:

[1] It has been found in the petroleum laboratory of the Mellon Institute of Industrial Research that agitation with successive small quantities of hot concentrated alkali tends to minimize the emulsification of treated lubricating oils.

Agitation with first portion of sulphuric acid.	1½ hrs
Deposition of acid	¾ hr.
Agitation with remainder of acid	1½ hrs.
Deposition of acid	2 hrs.
Washing	1½ hrs.
Agitation with alkali	1½ hrs.
Final washing	1 hr.

It is sometimes customary to add the acid in three portions instead of two. This would seem to be good practice, based on correct scientific principles.

Sun-Bleaching.—Illuminating and lubricating oils often may be improved in color by exposing them in thin layers to the rays of the sun. In other cases, however, the exposure has the opposite effect, the color becoming darker instead of lighter. It is sometimes advantageous to ascertain the action of light upon the refined products of a crude oil under study.

The Use of Florida Earth and Animal Charcoal.—Animal charcoal is not employed so widely as formerly for the bleaching or decolorization of petroleum products, its main use being in the treatment of semi-solid residues for the production of petrolatum. Fullers' earth and bauxite are the decolorizing media which now are favored by refiners.

For the bleaching of mineral oils Florida earth generally is preferred to other fullers' earths of the United States.[1] The grades vary between 15 to 30 mesh, 30 to 60 mesh, and 60 to 80 mesh. The grade chosen will depend upon the viscosity of the oil and the temperature at which the filtration is to be carried out. It is possible to select a grade of certain mesh and to filter the lighter products cold, the heavier ones hot. The amount of earth necessary to produce a given color will, of course, depend mainly upon the original color of the product. To illustrate, in large-scale practice 1 ton of earth serves to bleach from 10 to 30 barrels of oil.[2]

For experimental purposes in investigating the quantity of earth necessary to produce the best effect, it is recommended

[1] However, Californian earths now are being used after receiving a special chemical treatment. The treated earth (Death Valley "clay" is the most efficient) is agitated with the oil at a temperature of 300°F., and the product then is filtered. The decolorizing power of treated "clays" of this type declines gradually with time and exposure.

[2] On the preparation of Florida earth prior to use and on the regeneration of used earth, see Bacon and Hamor, *lib. cit.*, **2**, 614–20.

that the lighter oils be filtered through 5 to 20 per cent. by weight and the darker ones through 30 per cent. or more by weight of dehydrated Florida earth. The filtrate is separated into 5 per cent. fractions as it comes from the filter. The colors of the fractions are determined, and finally the fractions are blended successively and the colors are ascertained at each step.

The Use of Cupric Oxide and Sodium Plumbite.—As a means of removing sulphur compounds, cupric oxide in excess is agitated with the oil during distillation[1] (Frasch process), before the final refining with sulphuric acid. While copper oxide removes the sulphur compounds more completely from the crude fractions, sodium plumbite treatment, which is applied after the final refining with sulphuric acid, is favored because of the simplicity of the process. The oil merely is agitated with the plumbite solution, after which it is customary to add a small quantity of ordinary flowers of sulphur to bring down the dispersed material in the body of the oil.[2] In the latter procedure it should be noted that some of the sulphur may go into solution.

4. Theory of Refining.—Crude petroleum is a complex of liquid hydrocarbons—mainly a solution of hydrocarbons in one another. Sulphur, oxygen and nitrogen compounds also may be present, as well as asphalt and sometimes paraffin in dispersed condition. The darker the oil, the greater the number of dispersed asphaltic particles.

When crude oil is dry-distilled, the components of lowest boiling-point come over into the condenser first, followed by the ones of higher boiling-point as the temperature rises. During the distillation, from the point where the first gases appear, to the 325°C. (617°F.) point, the products which are obtained are in the

[1] The maximum quantity of cupric oxide used is 6 pounds per barrel of distillate treated.

[2] Schwartz and Nevitt (*Petroleum*, **7** (1919), 23, 96, 98, 100 and 102) state that 30°Bé. sodium hydroxide solution dissolves about 3 per cent. of lead oxide (litharge). Theoretically, 0.6 gal. of this saturated solution is required for 0.01 per cent. of sulphur in one barrel of oil; but, in practice, three times the theoretical amount should be agitated with the oil, followed by the addition of powdered sulphur, then by settling, washing with water and with dilute sulphuric acid, and finally by neutralizing with dilute sodium hydroxide solution. In American refinery operations, it is customary to use sufficient "doctor" solution to "kill" the "sulphur," *i.e.*, until the oil under treatment no longer responds to the "doctor test." See page 59.

main *natural*, that is, they exist in the original crude oil as such. Above this temperature, however, *cracking* becomes evident by the stream from the condenser becoming darker and the odor of the gases evolved becoming more disagreeable. *Cracking*, or *pyrolysis*, is a property which organic compounds have of decomposing when heated at higher temperatures, to form coke and compounds (gases and liquids) of lower molecular weight and boiling-point. In the case of crude petroleum this cracking takes place mainly at the expense of the lubricating fraction.

When steam is injected into the oil at the bottom of the still, the residue is kept well agitated, thus preventing local superheating, and, what is more important, the fractions come over at a lower temperature than would be the case in dry distillation.[1] The result is that the lubricating fractions are of better quality.

Heavy asphaltic bodies which may be present in the crude oil, being of high molecular weight and consequently high boiling-point, do not come over during the distillation, but remain in the residue. Other impurities, such as oxygen and sulphur compounds, also tend to concentrate in the residue, but may also distil over or break down, giving off water and hydrogen sulphide. Molecular sulphur present in the original crude will react with the oil at temperatures above 200°C. (392°F.), giving unsaturated products which polymerize more or less, hydrogen sulphide being formed during the reaction.

The Action of Sulphuric Acid on Petroleum Distillates.—Sulphuric acid acts both chemically and physically upon petroleum. The physical action consists mainly in the solution of tar and other dispersed material in the acid, and the subsidence of it by its superior weight to the oil. Chemically the reaction with olefines comprises polymerization to dipolymers and the formation of esters and hydrated esters of sulphuric acid, the latter on treatment with water forming secondary and tertiary alcohols.[2] The odor of acid tars resembles that of certain secondary and tertiary alcohols. According to Brooks and Humphrey, the pure olefines do not have a disagreeable odor. The malodor of

[1] Liquids boil when the vapor-pressure slightly exceeds that of the atmosphere. In the case of immiscible liquids the total pressure is the sum of the partial pressures, so that the boiling-point is that temperature at which the sum of the partial pressures equals that of the atmosphere.

[2] Brooks and Humphrey, *J. Am. Chem. Soc.*, **40** (1918), 822.

unrefined distillates is to be attributed more to the presence of small amounts of naphthenic acids and organic nitrogen compounds.

Oxygen and sulphur compounds also are acted on to some extent by sulphuric acid, the completeness of the reaction depending upon the duration of the treatment and on the temperature.

The dipolymers formed by the action of sulphuric acid are very viscous, and, according to Brooks and Humphrey, are likely to find their way into the oil layer because they are slightly attacked or dissolved by sulphuric acid.

Action of Florida Earth.—Florida earth removes the coloring matter from mineral oils and also holds back the olefines and impurities. The first runnings from the filter are lower in flash-point and viscosity and lighter in gravity and color than those following.[1]

The efficiency of the bleaching action of fullers' earth on oils is due not to the chemical composition of the earth, but to its selective power of adsorption or capillary action.[2]

[1] On the use of fullers' earth for bleaching mineral oils, see VOLLERTSEN, *Cotton Oil Press*, **2** (1919), No. 11, 37; SIEGFRIED, *Nat. Pet. News*, **11** (1919), No. 34, 38; and PHALEN, Bureau of Mines *Mineral Investigations*, Aug., **1919.**

[2] PARSONS, *J. Am. Chem. Soc.*, **29** (1907), 598; PHALEN, *ref. sup.*

CHAPTER II

THE EXAMINATION OF PETROLEUM NAPHTHA PRODUCTS

I.—Motor Gasoline.

1. Color, Odor, Acidity, Water and Dirt.
2. Specific Gravity and Baumé Gravity:
   Hydrometer, Pycnometer and Westphal Balance.
3. Volatility.
4. Other Tests on Gasoline:
   (*A*) Loss to Sulphuric Acid.
   (*B*) Iodine Number.
   (*C*) Sulphur:
   Lamp Method and "Doctor" Test.
   (*D*) Heat of Combustion.
   (*E*) Corrosion and Gumming Test.
   (*F*) Spontaneous Ignition Temperature.
   (*G*) Composition.
   (*H*) Vapor Pressure.
5. Rules for the Shipment of Gasoline.
6. Gasoline Specifications.
7. Results of Tests.

II.—Petroleum Ether, Solvent Gasoline, and Cleaning Oil.

I. MOTOR GASOLINE ("GASOLENE")[1]

1. Color, Odor, Acidity, Water and Dirt.—The inspection for color, odor, water and dirt may be carried out by examination of the product in an oil-sample bottle of colorless glass. In some

[1] On the types of gasoline, see Bacon and Hamor, *lib. cit.*, **2**, 870. The word "petrol" is used in England as the general designation for motor gasoline; "petrol" in French is equivalent to the English "paraffin oil" (kerosene), while motor gasoline is commonly known in France as "essence." "White spirit" is a European motor fuel which is prepared by mixing benzol with 25 per cent. of petrol (English). On the European continent, the family name for all the light petroleum distillates is benzine (=American term *naphtha* and English term *spirit*). On German aviation petrols, see *Compt. rend.*, **169** (1919), 693. Twenty-two samples approximated the composition: aromatic hydrocarbons, 10 per cent.; saturated cyclic hydrocarbons, 40 per cent.; and acyclic hydrocarbons, 50 per cent. The method of analysis was based on the critical temperature of solution in aniline (see *J. Soc. Chem. Ind.*, **38** (1919), 619A and 672A.).

cases it may be necessary to make a determination of the color by means of a colorimeter, as described under illuminating oils on page 88 (see also the *Appendix*, page 353).

Acidity Test. *Apparatus and Reagents.*—Oil-sample bottle; distilled water; and blue litmus paper.

Procedure.—Ten c.c. of the gasoline are shaken thoroughly with 5 c.c. of distilled water and the separated water is tested for acidity with litmus paper. If the litmus paper turns red, the test should be reported as positive; the acid present may be determined quantitatively, as described for the determination of acidity under *lubricating oils* (see page 96). If there is no reaction with the litmus, a negative test is reported.

As motor gasoline itself seldom contains acids, it is recommended that the residue left in the flask after the completion of the analytical distillation be shaken with 1 c.c. of distilled water and the separated water tested for acidity. The presence of acid in the residue after distillation may be caused by the decomposition of sulphuric acid derivatives. "Pyrolytic" gasolines which have not been redistilled after sulphuric acid treatment may display this property.[1]

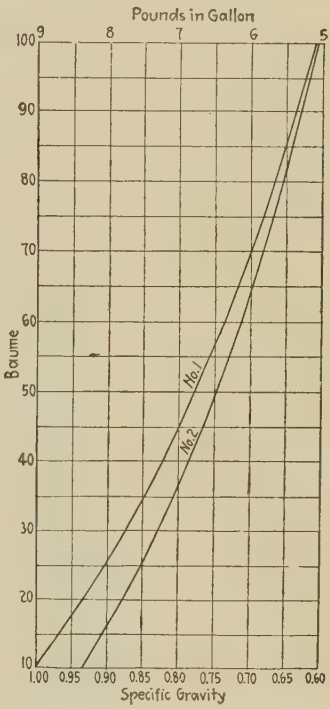

Fig. 18.—Chart illustrative of the relation of Baumé degrees to specific gravity (curve No. 1) and the weight of one U. S. gallon at 60°F. (curve No. 2).

2. Specific Gravity and Baumé Gravity.—For convenience and rapidity in determining the specific gravity, the operation is carried out at the temperature of the laboratory and corrected to $\frac{60°F.}{60°F.}$ by means of the correction tables given in the *Appendix*, pages 304 and 312.

The Baumé degrees are based on the modulus 140, and the

[1] See Bureau of Mines *Tech. Paper* **214.**

equation connecting Baumé degrees and specific gravity is as follows:

$$Sp.\ gr.\ 60°/60° = \frac{140}{130 + Bé.°_L}.$$

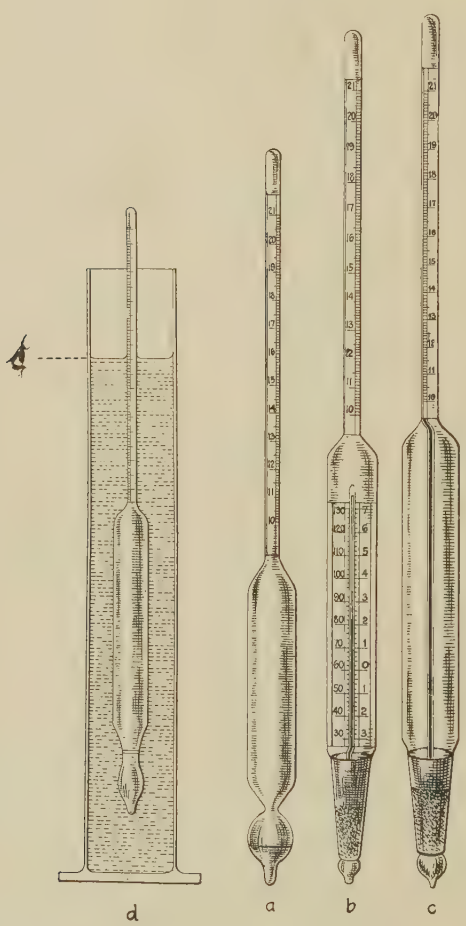

FIG. 19.—The Baumé hydrometer. (*a*, *b* and *c* illustrate forms of instruments in use, and *d* presents the application.)

As the Baumé scale for light liquids is used universally in the petroleum industry, it will be adhered to in this work. Con-

[1] TABER (*loc. cit.*) uses the abbreviation Bé.°$_L$ when referring to the gravity of liquids lighter than water; likewise Bé.°$_H$ is suggested for liquids heavier than water: Sp. gr. 60°/60° $= \frac{145}{145 - Bé.°_H}$.

versions may be made by means of the above equation or by means of the *Appendix*, page 304. All determinations in laboratory reports, except in the case of asphalt, are to be reported in the Baumé scale.[1]

Method A. Hydrometric Method (see Fig. 19). *Apparatus.*—Thermometer (provided the hydrometer does not include one[2]); Baumé hydrometer; and 500-c.c. cylinder.

Procedure.—The gasoline is poured into the cylinder, and the hydrometer and thermometer then are inserted, the former being allowed to float, while the latter is suspended in the gasoline one-half way down the cylinder. After fifteen seconds, readings of the hydrometer and thermometer are taken and recorded. The hydrometer reading should be made directly on the meniscus for clear oils, while, for dark oils, the surface is read and corrected by a predetermined constant.

If the temperature of the gasoline is other than 60°F., the observed Baumé reading must be corrected to 60°F. by means of the *Appendix*, page 304.

Note.—Where speed is not a requisite in using the Baumé hydrometer, it is practicable, and also more accurate, to make the determination with the liquid at a temperature of 60°F. exactly. A convenient method for doing this is first to place the cylinder in water at a temperature of 60°F. and, after cooling the gasoline to a temperature of 60°F., to pour it into the cylinder and then to insert the hydrometer. If the temperature of the water has risen in the meantime, pour in ice-water until the temperature is again at 60°F. Finally, stir the gasoline until it is at 60°F. and, after a few seconds, read the hydrometer.

Method B. Pycnometric Method. *Apparatus.*—Pycnometer (Fig. 20) and analytical balance.

Procedure.—Carefully clean and dry the pycnometer and then weigh it. Fill with distilled water at a definite temperature (or proceed with the aid of a water-bath, as described in the following paragraph) and weigh again, being sure that the capillary is entirely filled, that there are no air bubbles mixed with the water, and that the outside of the pycnometer is clean and dry. Read the temperature of the thermometer just after filling the

[1] Specific gravity may be included advantageously.

[2] A thermohydrometer, a modified form of hydrometer in which a thermometer is situated inside the float, is a useful instrument which enables a simultaneous reading of both temperature and Baumé gravity.

pycnometer. Knowing the weight and temperature of the water in the pycnometer, calculate the "water equivalent" of the pycnometer as follows: Multiply the weight of water found by the density of water at 60°F. (15.56°C.) and divide by the density of the water at the temperature at which it occurs in the pycnom-

Fig. 20.

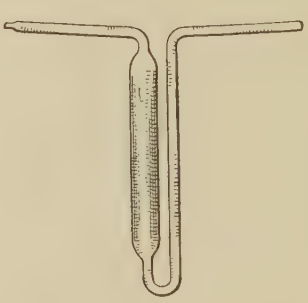

Fig. 21.

Fig. 20.—Specific gravity bottle, with ground-in thermometer and stoppered capillary tube.

Fig. 21.—Nicholl's specific gravity tube. A similarly convenient and simple apparatus is the well-known Sprengel tube, a variety of pycnometer which has expanded to form a double body both branches of the U-shaped tube. On the Ostwald modification, see Ostwald's "Physico-Chemical Measurements" (trans. by Walker), 1894.

eter. The following table presents the density of water at various temperatures:

Temperature °C.	Density.	Temperature °C.	Density.
14	0.999277	23	0.997568
15	0.999132	24	0.997326
16	0.998976	25	0.997073
17	0.998808	26	0.996811
18	0.998628	27	0.996540
19	0.998437	28	0.996260
20	0.998235	29	0.995971
21	0.998023	30	0.995674
22	0.997800		

Next clean and dry the pycnometer carefully, fill it with the oil and keep at room or other desired temperature in a water-bath until the temperature is constant.[1] It then is weighed, observing the same precautions as in the water weighing. Note

[1] Where a number of determinations of specific gravity have to be made, it is convenient to use a bath, the temperature of which is maintained constant by an automatic thermo-regulator. For a *rapid* method, see page 127.

the temperature of the oil at the time the pycnometer is filled. Then:

$$\text{Specific gravity}\frac{t°C.}{15.56°C.} = \frac{\text{weight of oil at } t°C.}{\text{water equivalent.}}$$

This value now must be corrected to $\frac{60°F.}{60°F.}\left(\frac{15.56°C.}{15.56°C.}\right)$, by the data given in the *Appendix*, page 304, first changing the value for specific gravity to Baumé gravity.

Other Pycnometers.—Where but very small samples are available, a pycnometer of the type shown in Fig. 21 may be used. The procedure is similar to that employed in the above method, except that the sample and the water must be exactly at the room temperature and their temperatures read before filling the tube,

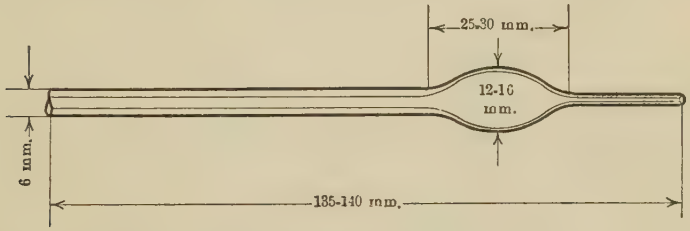

FIG. 22.—Drushel pycnometer of dimensions and construction suitable for measuring specific gravities of petroleum distillates. This instrument is useful in the examination of very small amounts of products.

which should not be touched with the hands. After the tube has been filled, the level of the liquid on the side having the mark is adjusted exactly to the mark, by drawing off some of the liquid by means of a roll of filter paper.

METHOD C. WESTPHAL BALANCE METHOD. *Apparatus.*—Westphal specific gravity balance and accessories (see Fig. 23).[1]

Procedure.—See that the plummet is clean and dry. Place it on the hook at the end of the beam, and, by means of the ad-

[1] The complete instrument consists of the balance proper, the glass plummet, the immersion cylinder, and two exactly similar sets of four rider weights, the largest rider of each set having the same weight as the water which the plummet displaces at 60°F., the next rider having one-tenth of this weight, the third one-hundredth, and the fourth one-thousandth. The beam is divided by notches into ten exactly equal parts, the notches being points of suspension for the riders and numbered consecutively from 1 to 9. The specific gravity "chainomatic" balance provides another method for obtaining direct readings in specific gravity to the third or fourth decimal place.

justing weight, regulate the swing of the beam so that the pointer moves equal distances above and below the zero mark on the scale. The apparatus may be checked further by immersing the plummet in water at 60°F. (15.56°C.), when the largest rider or horseshoe weight, suspended on the hook at the end of the beam, should bring it to equilibrium.

The liquid to be tested is poured into the cylinder and the plummet is immersed and attached to the hook. The plummet should be immersed so that, on the upward swing, it does not come above the surface. By means of the horseshoe or rider weights placed in the notches of the beam, the beam is brought to equilibrium with the pointer opposite the zero mark. Thus, the specific gravity of the liquid is shown at a glance to four decimal places. The largest weight represents tenths, and so gives the first decimal place; the position of it relative to the center of the beam gives the number of tenths. The other riders give the remaining decimal places in order: the next smaller weight represents hundredths, the next thousandths, and the next ten-thousandths.

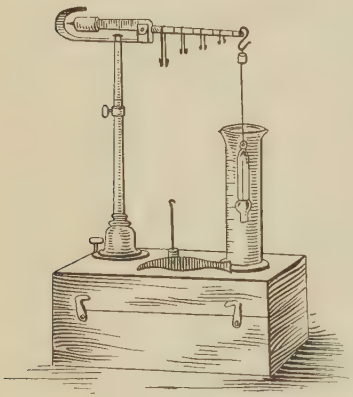

FIG. 23.—The Westphal balance.

The temperature of the sample should be taken at the time of the determination; and if it is other than 60°F. (15.56°C.), the specific gravity should be corrected to 60°F. The liquid at the time of the determination may be taken conveniently at 60°F. by first bringing the liquid to 60°F. and pouring it into the cylinder, which is surrounded by water kept at the temperature of 60°F.

3. Volatility.—This test may be considered the most important one for motor gasoline. The principle of the method, that of distillation, is simplicity itself, and this fact alone is conducive to neglect of the details which must be observed in order to secure concordant and true results.

Motor gasoline may be looked upon as being composed of three portions as regards volatility: (1) A low-boiling, volatile portion, which is of value in starting; (2) an intermediate portion, or

body; and (3) the power portion,[1] which is desirable in as large a percentage as is commensurate with the ability of the carbureter to volatilize it so that it will burn completely. The power portion is the highest boiling of the three, but should not be of so high a boiling-point that an excessive amount of carbon is produced in the cylinders. Therefore, it is evident that, all other things being equal, the best gasoline is the one which has the proper blending[2] of the three portions.

For the study of distillation curves, a distillation method, such as Method A, may be supplemented by a method whereby a fractionating column is used (Method B). By the use of the last-mentioned method the three portions described above will be brought out better when the distillation curve is drawn. For examples of distillation curves, see the *Appendix*, pages 334 and 352. For a blending curve, see the *Appendix*, page 345.

The method of distillation adopted by the U. S. Fuel Administration is described below; it is probably as nearly standard as any now in use, although other methods are employed to a large extent.[3] Often a 200-c.c. sample is used in a large flask, with

[1] There is the belief that the use of motor gasoline containing "high (boiling) ends" results in a decrease in carbonization troubles, because of a cleansing action within the cylinders; but that there is leakage past the piston rings, with consequent solution in and decrease in viscosity of the lubricating oil, necessitating the employment of more and heavier lubricating oil.

[2] By the term "blending" is meant the mixing of various petroleum products or fractions to give a *homogeneous* product, *i.e.*, one in which the components are completely miscible with one another by inherent characteristics and have been made so by proper mechanical agitation or method of mixing. A properly or "scientifically" blended gasoline is one which has been secured by the mixing of "straight-run" gasoline with natural-gas gasoline (casinghead, absorption gasoline, etc.) and possibly naphtha and "cracked" gasoline in the right proportions. Whether or not a gasoline has been properly "blended" can be found by distilling a sample (Method B) and plotting the distillation curve. Faulty blending then can be detected by a study of this curve.

[3] The following summary is pertinent concerning the standardization of distillation tests for motor gasoline:

The multiplicity of methods which have been proposed for testing motor gasoline by distillation leads generally to much difficulty and inconvenience when it is desired to compare the figures of tests made on products from the same or different sources which have been reported by different analysts. It is not of much importance whether a given method of distillation gives rather lower or higher values for any given fraction. What is of the greatest importance, however, is that the method used shall be capable of giving as

or without the flask resting on a sand-bath during the distillation. The only objection to the sand-bath is the difficulty experienced in determining the end-point. The effect of increasing the volume of the sample distilled is to lower the over-point.

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closely concordant results as possible on repeat tests when made by the same or different observers. The method also should be as rapid, simple, and easy to carry out as the nature of such a test permits.

In 1904, H. S. Garry and H. J. Watson (*J. Soc. Chem. Ind.*, **23** (1904), 701) called attention to the necessity for a standardized test for petroleum spirits similar to that adopted for benzene. This standardization has not so far been achieved, but has recently been strongly advocated by A. Duckham (*J. Inst. Pet. Tech.*, **4** (1917), 4) and other prominent members of the Institution of Petroleum Technologists. It is hoped that action shortly will be taken to decide upon a generally acceptable method of test. It is not necessary that the selected method shall embody all the details of any test now in use. It may and probably will be arrived at finally by a judicious selection and combination of details of several or of all these processes. It is somewhat remarkable that of two of the most recent and important papers on methods of testing gasoline by fractionation (Lomax, *J. Inst. Pet. Tech.*, **4** (1917), 6; and Anfilogoff, *J. Soc. Chem. Ind.*, **37** (1918), 21T.), neither gives the results of a series of repeat tests on one sample of spirit. Such a series of tests is always necessary in order to exhibit the variations in the results, which are to be expected from experimental errors inherent in any particular method. Attention was called to this omission by Cadman in the course of discussion on the paper by Lomax (*J. Inst. Pet. Tech.*, **4** (1917), 34). All the results of tests given were, it is true, the mean values of four or five separate tests, but the results of these separate tests were not stated.

Lomax states that the liability to personal error is much greater with the Engler method than with the Redwood, great care being required even by skilled manipulators to obtain concordant results on the same sample of spirit, while independent operators may obtain results varying within comparatively wide limits. Most investigators fully concur in this opinion. Experience is that Engler's method is unworkable unless a very low standard of agreement of duplicate tests is to be accepted. In spite of this fact some chemists continue to compare their methods with the results of tests obtained by the Engler method. This practice, no doubt, is followed only because the Engler method possesses a long-standing and somewhat exaggerated text-book reputation.

A novel form of apparatus for fractioning motor gasoline and other volatile liquids for laboratory testing purposes is described by E. Hildt (*Compt. rend.*, **165** (1917), 790). It consists of a series of glass receivers each connected with its neighbor on its right and left by a tube at the top, and each heated to a fixed temperature higher than that of the next receiver. The spirit to be fractionated is admitted drop by drop into the receiver at the highest temperature. Here it is completely or partially vaporized, and the vapor flows along the tubes through the series of receivers until it arrives

METHOD A. U. S. BUREAU OF MINES METHOD (1917), SLIGHTLY MODIFIED; based upon the procedure of the American Society for Testing Materials.[1]

Apparatus (see Fig. 24).—Standard Engler flask; 300°C. (572°F.) standardized thermometer; trough condenser; burner; asbestos board, 4 in. square, having a 1¼ in. hole in the center; 100-c.c. measuring cylinder; and auxiliary thermometer for use in case the thermometer is calibrated for total immersion.

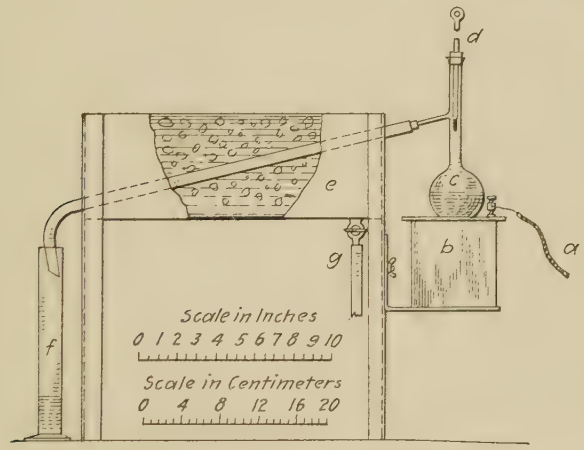

FIG. 24.—Apparatus used by the Bureau of Mines for distillation test of gasoline.
a, Wires connecting with electric mains through a suitable rheostat. *b*, Electric heater. *c*, Engler distillation flask filled with charge of gasoline partly distilled. *d*, Thermometer. *e*, Condenser, with trough filled with ice and water. *f*, Receiving graduate. *g*, Cock for draining condenser trough.

Procedure.—The condenser is first filled one-fourth full with cracked ice, and the remainder with ice-water. 100 c.c. of the gasoline are measured into the cylinder, drained into the flask, and the thermometer and cork are inserted. The thermometer should have a film of cotton wrapped about the bulb to assist in preventing fluctuations of temperature. The bulb should be

at one in which the temperature is sufficiently low for it to condense. This method of fractionation is so novel for laboratory testing that it will be of interest to have details of extended trials, especially as it appears to permit of a distillation curve being obtained at one operation over a considerable temperature range, the fractions being collected at any desired intervals.

On testing motor spirits, see also PHILIP, *Pet. Times*, **2** (1919), 401, 429 and 451.

[1] Bureau of Mines *Tech. Paper* **166**, by E. W. DEAN.

located just below the outlet tube and exactly in the center of the cross-section of the neck of the flask. The cylinder is placed at the outlet end of the condenser, and, in case a very light product is being distilled, should itself be immersed in ice-water with a small piece of cardboard on top, through which runs the outlet tube from the condenser. Three or four small grains of sand in the flask with the gasoline will assist to prevent overheating and subsequent bumping.[1]

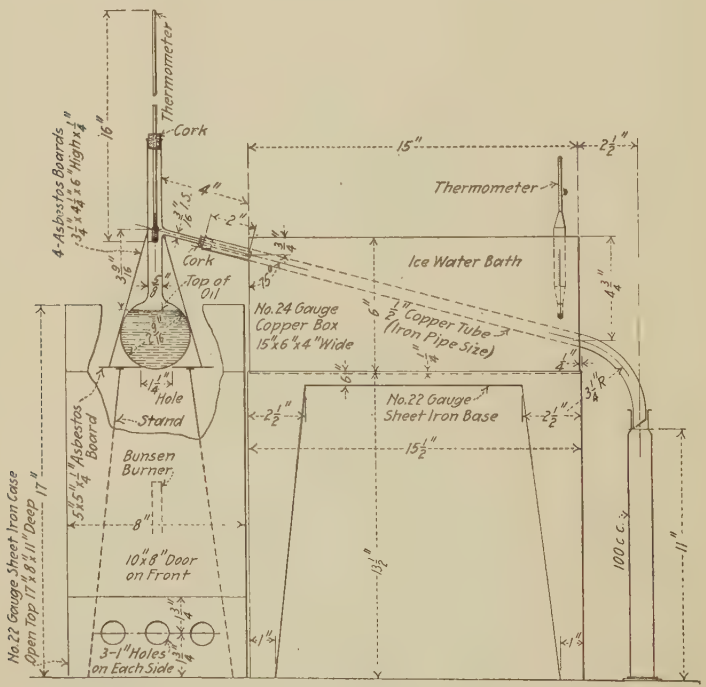

FIG. 25.—Modified Bureau of Mines apparatus for distillation, as used by Cosden and Company.

In starting the distillation care should be taken that the flask is not heated too strongly. The distance of the tip of the flame from the bottom of the flask should depend mainly upon the volatility of the product and should be so regulated that the distillation starts at the proper rate. The ability to do this is acquired through practice. In order to have a constant rate of distillation, the flame of the burner should be surrounded by a shield,

[1] A small piece of porous plate also will serve this purpose.

and the entire operation is carried out preferably in a cabinet, in order to eliminate draughts. When the first drop falls from the condenser into the cylinder, the temperature is read and is recorded as the *over-point.* The distillation then should proceed at the rate of 4 to 5 c.c. per minute, and the flame should be increased slowly and continuously in such a way that the proper rate is held throughout the distillation.[1]

For each 10 c.c. accumulating in the receiver a reading of the thermometer is made and recorded. The highest temperature reached after the flask goes dry, is read and recorded as the *end-point.* The last reading of the distillate is not made until the vapors remaining in the flask have condensed and cooled and have been poured into the receiver. The final reading of the distillate subtracted from 100 gives the *distillation loss* (*cf.*, however, page 56).

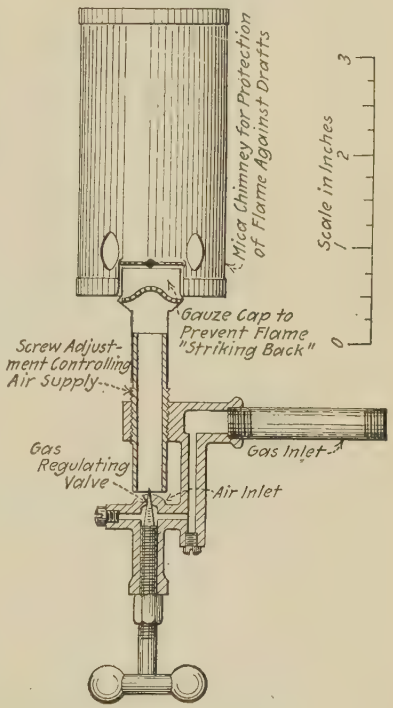

FIG. 26.—Special gas burner designed and employed for gasoline distillation by the Atlantic Refining Company. It is composed of a special bronze casting equipped with sensitive needle-valve and a screw adjustment for the regulation of the air-gas mixture.

During the course of the distillation several readings of the air-temperature at the center of the exposed portion of the thermometer stem, are made and recorded, provided, of course, the thermometer is one that has been calibrated at total immersion. Special thermometers are made for Engler flasks which are calibrated so that no correction of the observed boiling-point is necessary, this being a matter which should be determined before the disillation is begun.

[1] The rate of heating before distillation begins is under investigation. It is thought that more consistent over-points may be secured by heating the gasoline in the flask so that it comes to the boiling-point in a definite length of time after applying the heat.

Finally, the corrected temperatures corresponding to the 10 per cent. distillates, along with the end-point and over-point, are plotted against the percentages distilled. The line through the points is the distillation curve.

A reading of the barometer should be made either just before or after the distillation.

Notes and Precautions.—In carrying out the distillation it is desirable that the burner used be of such design that the flame may be increased slowly and continuously until the end of the distillation. For this purpose a burner such as that described by Dean,[1] and developed by the Atlantic Refining Company, is very satisfactory (see Fig. 26).

The end-point[2] is the highest temperature reached during the distillation and does not mean the temperature at which fumes appear in the flask. With certain gasolines, toward the latter part of the distillation, the temperature may drop suddenly before the flask is dry. Upon stronger heating, the boiling-point rises to the true end-point of the product. This phenomenon is caused by the presence of a small percentage of heavy oil in the gasoline.

An equation which is used largely for correcting temperatures observed on thermometers which have been standardized while totally immersed, is as follows:

$$V = 0.00015^{3}\ (T - t)\ n.$$

V = correction to be added to observed reading.
T = observed boiling-point.
t = auxiliary temperature.
n = length of mercury column above vapor expressed in scale divisions.

The equation for variation of atmospheric pressure from normal is as follows:

$$V = 0.00012\ (760 - p)\ 273 + t.$$

V = correction to be added to observed reading.
p = observed pressure in millimeters.
t = the observed temperature in degrees C.

[1] *J. Ind. Eng. Chem.*, **10** (1918), 823.

[2] Sometimes designated *final* or *maximum boiling-point.* The *dry-point* is secured by reading the thermometer just as the flask becomes dry.

[3] This coefficient, which varies between 0.00015 and 0.00016, is for readings of the Centigrade scale. For Fahrenheit readings the coefficient ranges from 0.000083 to 0.000089.

Specifications for distillation thermometers are given as follows by Dean: [1]

"The thermometer should be made of selected enamel-backed tubing having a diameter between 5.5 and 7 mm. The bulb should be of Jena normal or Corning normal glass; its diameter should be less than that of the stem and its length between 10 and 15 mm. The total length of the thermometer should be approximately 380 mm. The range should cover 0°C. to 270°C., with the length of the graduated portion between the limits of 210 to 250 mm. The point marking a temperature of 35°C. should be not less than 100 mm. nor more than 120 mm. from the top to the bulb.

"The scale should be graduated for total immersion. The accuracy must be within about 0.5°C. The space above the meniscus must be filled with an inert gas, such as nitrogen, and the stem and bulb must be thoroughly aged and annealed before being graduated.

"All material and workmanship must be of the best. The scale shall be marked for single-degree intervals. Each tenth-degree shall be numbered and each fifth-degree distinguished by a longer mark."

The Committee on Standardization of Petroleum Specifications of the United States Fuel Administration recently has adopted the following method of test and specifications for gasoline, upon the recommendation of the Bureau of Mines and other authorities; it will be noted that the procedure differs merely in details from Method A, *supra*.

Quality.—Gasoline to be high grade, refined, and free from water and all impurities, and shall have a vapor tension not greater than 10 lbs. per sq. in. at a temperature of 100°F., same to be determined in accordance with the current "Rules and Regulations for the Transportation of Explosives and Other Dangerous Articles by Freight," as issued by the Interstate Commerce Commission.

Inspection.—Before acceptance the gasoline will be inspected. Samples of each lot will be taken at random. These samples immediately after drawing shall be retained in a clean, absolutely tight, closed vessel and a sample for test taken from the mixture in this vessel directly into the test vessel.

Specifications.

(*a*) Boiling-point must not be higher than 60°C. (140°F.).
(*b*) 20 per cent. of the sample must distil below 105°C. (221°F.).
(*c*) 50 per cent. must distil below 140°C. (284°F.).

[1] Bureau of Mines *Tech. Paper* **214.**

(*d*) 90 per cent. must distil below 190°C. (374°F.).

(*e*) The end- or dry-point of distillation must not be higher than 225°C. (437°F.).

(*f*) Not less than 95 per cent. of the liquid will be recovered in the receiver from the distillation.

One hundred c.c. will be taken as a test sample. The apparatus and method of conducting the distillation shall be that adopted by Sub-Committee XI of Committee D-1 of the American Society for Testing Materials, with the following modifications:

First, the temperature shall be read against fixed percentage points; and second, the thermometer shall be as hereinafter described.

Flask.—The flask used shall be the standard 100-c.c. Engler flask, described in the various textbooks on petroleum. Its dimensions are as follows:

Dimensions	Cm.	In.
Outside diameter of bulb	6.5	2.56
Outside diameter of neck	1.6	0.63
Length of neck	15.0	5.91
Length of vapor tube	10.0	3.94
Outside diameter of vapor tube	0.6	0.24

Position of vapor tube, 9 cm. (3.55 in.) above the surface of the gasoline when the flask contains its charge of 100-c.c. The tube is approximately in the middle of the neck. The observance of the prescribed dimensions is considered essential to the attainment of uniformity of results. The flask shall be supported on a ring of asbestos having a circular opening, 1¼ in. in diameter; this means that only this limited portion of the flask is to be heated. The use of wire gauze is forbidden.

Condenser.—The condenser shall consist of a thin walled tube of metal (brass or copper), ½ in. in internal diameter and 22 in. long. It shall be set at an angle of 75 degrees from the perpendicular and shall be surrounded with a cooling jacket of the trough type. The lower end of the condenser shall be cut off at an acute angle and shall be curved down for a length of 3 in. The condenser jacket shall be 15 in. long.

Thermometer.—The thermometer shall be made of selected enamel-backed tubing having a diameter between 5.5 and 7 mm. The bulb shall be of Jena normal or Corning normal glass; its diameter shall be less than that of the stem and its length between 10 and 15 mm. The total length of the thermometer shall be approximately 380 mm. The range shall cover 0°C. (32°F.) to 270°C. (518°F.), with the length of the graduated portion between the limits of 210 to 250 mm. The point marking a temperature of 35°C. (95°F.) shall not be less than 100 mm. nor more than 120 mm. from the top of the bulb. For commercial use the thermometer may be graduated in the Fahrenheit scale. The scale

shall be graduated for total immersion. The accuracy must be within about 0.5°C. The space above the meniscus must be filled with an inert gas, such as nitrogen, and the stem and bulb must be thoroughly aged and annealed before being graduated.

Source of Heat in Gasoline Distillation.—The source of heat in distilling gasoline may be a gas burner, an alcohol lamp or an electric heater.

Procedure and Details of Manipulation.—(1). If an electric heater is used it is started first to warm it. (2). The condenser box is filled with water containing a liberal portion of cracked ice. (3). The charge of gasoline is measured into the clean, dry Engler flask from a 100 c.c. graduate. The graduate is used as a receiver for distillates without any drying. This procedure eliminates errors due to incorrect scaling of graduates and also avoids the creation of an apparent distillation loss due to the impossibility of draining the gasoline entirely from the graduate.

(4). The above mentioned graduate is placed under the lower end of the condenser tube so that the latter extends downward below the top of the graduate at least 1 in. The condenser tube should be so shaped and bent that the tip can touch the wall of the graduate on the side adjacent to the condenser box. This detail permits distillates to run down the side of the graduate and avoids disturbance of the meniscus caused by the falling of drops. The graduate is moved occasionally to permit the operator to ascertain that the speed of distillation is right, as indicated by the rate at which drops fall. The proper rate is from 4 c.c. to 5 c.c. per minute, which is approximately two drops a second. The top of the graduate is covered, preferably by several thicknesses of filter paper, the condenser tube passing through a snugly fitting opening. This minimizes evaporation losses due to circulation of air through the graduate and also excludes any water that may drip down the outside of the condenser tube on account of condensation on the ice-cooled condenser box.

(5). A boiling stone (a bit of unglazed porcelain or other porous material) is dropped into the gasoline in the Engler flask. The thermometer is equipped with a well fitted cork and its bulb covered with a thin film of absorbent cotton (preferably the long-fibered variety sold for surgical dressing). The quantity of cotton used shall be not less than 0.005 nor more than 0.010 g. (5 to 10 mg.). The thermometer is fitted into the flask with the bulb just below the lower level of the side neck opening. The flask is connected with the condenser tube.

(6). Heat must be so applied that the first drop of the gasoline falls from the end of the condenser tube in not less than five or more than ten minutes. The *initial boiling-point* is the temperature shown by the thermometer when the first drop falls from the end of the condenser tube into the graduate. The operator should not allow himself to be

deceived, as sometimes (if the condenser tube is not dried from a previous run) a drop will be obtained and it will be sometime before a second one falls; in this case the first drop should be ignored. The amount of heat is then increased so that the distillation proceeds at a rate of from 4 c.c. to 5 c.c. per minute. The thermometer is read as each of the selected percentage marks is reached. The *maximum boiling-point or dry-point* is determined by continuing the heating after the flask bottom has boiled dry until the column of mercury reaches a maximum and then starts to recede consistently.

(7). *Distillation loss* is determined as follows: The condenser tube is allowed to drain for at least five minutes after the heat is shut off, and a final reading is taken of the quantity of distillate collected in the receiving graduate. The distillation flask is removed from the condenser and thoroughly cooled as soon as it can be handled. The condensed residue is poured into a small graduate or graduated test-tube and its volume measured. The sum of its volume and the volume collected in the receiving graduate, subtracted from 100 c.c., gives the figure for distillation loss.

Method B. Fractionating Column Method. *Apparatus.*—See Fig. 27. The fractionating column shown may be replaced by any efficient column which drains well. The shield about the column aids materially in carrying out the distillation.

Procedure.—The same as in the case of Method A, except that the rate is 2½ c.c. per minute. The *end-point* is specified as that temperature at which fumes appear in the flask.[1]

4. Other Tests on Gasoline. (*A*) Loss to Sulphuric Acid. Concentrated sulphuric acid reacts with unsaturated hydrocarbons, and to some extent with oxygen and sulphur compounds, thus removing them from gasoline. The effect of sulphuric acid upon unsaturated hydrocarbons has been investigated carefully and the knowledge of the subject extended greatly by Brooks and Humphrey.[2]

[1] For a general discussion of this method, see Lomax, *J. Inst. Pet. Tech.*, **4** (1917), 6. A 14-in. fractionating column, filled with either aluminum or magnesium turnings, and covered with asbestos, has been found to give good results with this method.

[2] *J. Am. Chem. Soc.*, **40** (1918), 822. The Maumené test or "acid heat test" is a method largely used in laboratories of refineries for determining the degree of unsaturation. According to the Bureau of Mines (*Tech. Paper*, **181**, 22), this test has proved unreliable, especially for highly unsaturated mixtures. Primary objections are the many empirical factors involved and the dissipation of much of the heat in volatilizing gasoline instead of producing a rise in temperature. See Bureau of Mines *Tech. Paper* **181** for an accurate method.

Two methods for conducting the "loss to sulphuric acid" test are presented below. Method B has the advantage of the use of apparatus which can be found in almost any laboratory. It should be kept in mind that this method is not intended to show the refining loss in refinery operations, but is a procedure for removing the unsaturated hydrocarbons and other impurities from gasoline. It is employed mainly in recognizing a slightly refined cracked (or pyrolytic) gasoline which has been blended with a more saturated product. It may be noted here, however, that benzene-

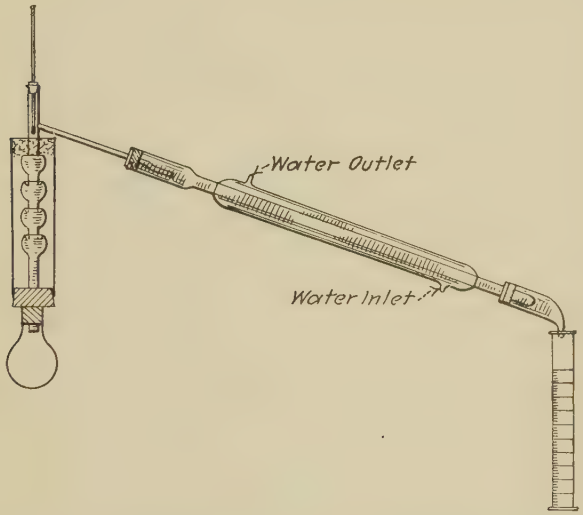

FIG. 27.—Lomax distillation apparatus. The innovation is the insulating shield about the column.

gasoline mixtures are in use as motor fuels, and that consequently the determination of the percentage of aromatic hydrocarbons is required rather frequently in practice.[1]

METHOD A. *Apparatus and Reagents.*—75-c.c. heavy-walled

[1] It may be mentioned that THOLE (*J. Soc. Chem. Ind.*, **38**, 49T.) has proposed the following method for estimating aromatic hydrocarbons in petroleum: A fraction of the proper boiling range first is prepared and then is treated with three times its volume of 98 per cent. sulphuric acid over a period of thirty minutes, with frequent shaking. Knowing the specific gravity of the aromatic component, the percentage present is calculated as follows:

Per cent. of aromatic = [(initial specific gravity − final specific gravity)/(specific gravity of aromatic − final specific gravity)] × 100.

COLMAN and YOEMAN (*ibid.*, 82T.) suggest the following equations in

flask, with a neck 50 cm. long and graduated to 0.1 c.c.; and c.p. sulphuric acid (1.84 sp. gr.).

Procedure.—25 c.c. of the gasoline are measured accurately into the flask from a pipette or burette, 25 c.c. of the sulphuric acid then are poured in, and the flask is stoppered and shaken vigorously for 15 minutes. After 30 minutes, sulphuric acid is added until the gasoline is forced into the neck. After standing overnight, the volume of the gasoline in the neck of the flask is read. This reading is subtracted from the original volume (25 c.c.), giving as a result the loss to sulphuric acid.

METHOD B.—U. S. BUREAU OF MINES METHOD.[1]

Apparatus and Reagents.—Babcock cream bottle;[2] burette or 5 c.c. pipette; c.p. sulphuric acid (1.84 sp. gr.); and centrifuge machine.

Procedure.—Five c.c. of the gasoline are run into a clean dry bottle from the burette or pipette, cooled for a minute or two by immersion in ice-water, and 200 per cent. by volume of sulphuric acid (1.84 sp. gr.) is added from a small graduate. A rubber stopper then is placed in the bottle and the contents are shaken, first slowly, then vigorously, with a rotary motion, for several minutes.

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calculating the percentages of aromatics from data secured by the sulphonation method of THOLE:

Benzene $= 100[(b_1 - k_1) - a_1]/(0.855 - a_1)$.

Toluene $= 100[(b_2 - k_2) - a_2]/(0.872 - a_2)$.

Xylene $= 100[(b_3 - k_3) - a_3]/(0.868 - a_3)$.

a_1, a_2 and a_3 are the respective specific gravities after removal of the benzene, toluene and xylene.

b_1, b_2 and b_3 are the respective specific gravities before sulphonation.

k is the amount by which the specific gravity of a mixture of that percentage of the aromatic hydrocarbon and gasoline is lowered by the expansion which occurs on mixing them. The above-cited authors give graphs for the values of k.

See also this Chapter, page 62; and Chapter IX, page 199. On benzol mixtures as motor fuels, see MACKIE, *Canadian Chem. J.*, **3**, 295.

The use of mixed motor fuels is still in its infancy, but mixtures have been adopted which show, under certain conditions, some advantages over motor gasoline. The British Air Forces use a mixture of 20 per cent. of benzol and 80 per cent. of petrol (see *Chem. Age* (Eng.), **2** (1920), 16). "Alcogas" is a mixture of 38 parts of ethyl alcohol, 19 parts of benzol, 4 parts of toluol, 30 parts of gasoline, and 7½ parts of ethyl ether (see Brit. Patents 128915, 128916 and 128917, of June 16, 1919). On benzene—gasoline—alcohol motor fuels, see *Chim. et Ind.*, **3** (1920), 761.

[1] *Tech. Paper* **181.**

[2] The neck should be graduated so that at least 5 c.c. may be read off.

The gasoline and sulphuric acid then are separated by adding sulphuric acid until the surface is about level with the upper graduation mark on the neck of the bottle, and allowing to stand overnight, when acid again is added until the gasoline meniscus is at the top graduation mark on the neck of the bottle. A reading of the volume of the gasoline remaining then is made, and the loss to sulphuric acid is calculated. Instead of permitting the bottle to stand overnight, it may be placed in a centrifuge (stoppered) and whirled at the rate of 500 to 1000 r.p.m. for two or three minutes.

(B) Iodine Number.—The iodine number represents the percentage of iodine absorbed. This value, like that of absorption by sulphuric acid, is an index to the extent of the unsaturation of the product, but it does not give the percentage of unsaturated compounds in the gasoline; in fact, absorption by sulphuric acid will come nearer to indicating the percentage of unsaturated compounds. According to Dean,[1] the iodine number can be found roughly from the loss to sulphuric acid, by multiplying the latter by 6.5.

The method of Wijs[2] generally is preferred for the accurate determination of the iodine number. The Hanus method, if properly applied, is, however, satisfactory.[3] On account of the volatility of gasoline, care should be taken in weighing out the sample (0.1–0.15 g. in 25 c.c. of iodine solution) for analysis that there is no loss by evaporation.

(C) Sulphur.—Sulphur in gasoline may be detected by the method given on page 8. It may be determined quantitatively by the method described by Lomax,[4] wherein a weighed quantity of the gasoline in solution in absolute ethyl alcohol is burned in a lamp of special construction and the products of combustion are drawn through a N/16 solution of sodium carbonate, 1 c.c. of which equals 0.001 g. of sulphur; finally the sodium carbonate solution is titrated with N/32 sulphuric acid (see Fig. 28).

Free sulphur in gasoline is detected during the corrosion and gumming test (see page 61).

The *"doctor" test*, which has been in use by the Standard Oil

[1] Bureau of Mines *Tech. Paper* **181.**

[2] See page 21.

[3] *Cf.* Radcliffe and Polychronis, *J. Soc. Chem. Ind.*, **35** (1916), 340; and Dean, *supra.*

[4] *J. Inst. Pet. Tech.*, **4** (1917), 6. On sulphur in gasoline, see *Francis, Science*, **52** (1920), 256.

Company since the introduction of the Frasch process, for the detection of certain types of decomposable sulphur compounds, was included in war-time specifications for aeroplane gasoline. It does not indicate the actual presence or absence of sulphur in gasoline, and, in fact, products that have been refined to pass this test may contain a larger percentage of sulphur than before treatment. But gasoline not complying with the "doctor" test is likely to decompose in storage with the development of a yellow color and an offensive odor. In addition, there is the possibility that gasoline sour to the "doctor" test may have been the cause of certain reported corrosion of metal parts of carbureters.[1] The details of the test follow.

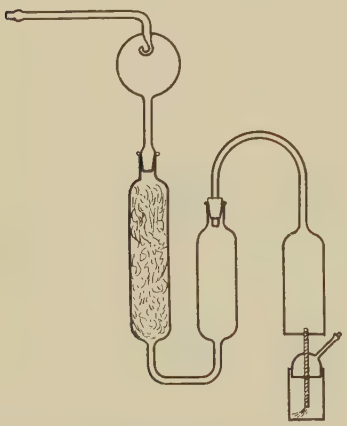

Fig. 28.—Lamp apparatus for the determination of sulphur.

Apparatus and Reagents.—Sodium plumbite, or "doctor" solution (prepared by dissolving 125 g. of sodium hydroxide in a liter of distilled water, adding 60 or 70 g. of litharge and shaking for 20 minutes, finally filtering through a mat of asbestos); flowers of sulphur; and test-tubes.

Procedure.—10 c.c. of gasoline and 5 c.c. of "doctor" solution are shaken together in a test-tube for 15 seconds. A small pinch of flowers of sulphur then is added, and the whole is agitated for 15 seconds more. Enough sulphur should be used so that the entire quantity floats on the surface separating the gasoline from the "doctor" solution. If the gasoline is discolored or if the sulphur film is so dark that the yellow color of the sulphur is noticeably masked, then the test should be reported as positive. If the sulphur film is bright yellow, or is only slightly discolored with gray or flecked with black, then the test should be reported as negative.

Many petroleum chemists prefer to determine the sulphur in gasoline as well as in other products by the method of combustion in a bomb with oxygen, the heat of combustion being found at

[1] On the principles of the "doctor" test, see Schwartz and Nevitt, *Petroleum*, **7** (1919), 23, 96, 98, 100 and 102.

the same time (see Chapter I, page 7). The main objection to the lamp method is the possibility of incomplete combustion of the sulphur compounds. However, it must be remembered that the lamp method has the advantage of the use of larger samples and of a form of apparatus which may be improvised readily from apparatus found in any laboratory.

(D) Heat of Combustion.—It is recommended that, wherever possible, this determination be carried out by means of the oxygen bomb, the sample being enclosed in a gelatin capsule of known heat value.

At this point it is of advantage that the distinction between *total* and *net heating values* be made. The total heat value is that found by the method of determination.[1] The net heat value is the datum which is of interest when considering practical combustion—in the internal combustion engine, for example, where the products are carried off at a temperature higher than 100°C. (212°F.). It may be defined as the total heat value minus the sum of the latent heat of vaporization at 100°C. of the amount of water (if any) present in the fuel, together with that of the further amount of water which is formed by its combustion.

(E) Corrosion and Gumming Test. *Apparatus and Reagents.*—Freshly polished, hemispherical dish of spun copper, approximately 3½ in. in diameter; and an analytical balance.

Procedure.—Weigh the copper dish, then fill it with gasoline to within ⅜ in. of the top, and evaporate on the water-bath until all volatile portions are gone; finally cool in a desiccator and weigh. It is specified that if the gasoline contains undesirable gum-forming constituents, there will be a weighable deposit left on the bottom of the dish; and if elementary sulphur is dissolved in the gasoline, the bottom of the dish will be colored black, giving a positive test. No discoloration or a gray or brown color is to be reported negative.

(F) Spontaneous Ignition Temperature.[2]—The test consists in allowing one drop of gasoline to fall on a platinum crucible heated to a desired temperature. If the temperature is

[1] When the determination is carried out in a bomb or calorimeter, the water in the sample, as well as that formed by combustion, gives up considerable heat when cooled to the temperature of the surrounding water. On calorimetric determinations, see page 503.

[2] Moore, *J. Soc. Chem. Ind.*, **36** (1917), 109; and Lomax, *J. Inst. Pet. Tech.*, **4** (1917), No. 13, 25. See also Moore, *Automobile Eng.*, **1918**, 245; and *J. Inst. Pet. Tech.*, **6** (1920), No. 22, 186.

much higher than the ignition-point, a sharp explosion takes place almost immediately; if below the ignition point, no explosion will occur. The lowest temperature at which an explosion takes place is called the *spontaneous ignition temperature.* This temperature is of interest to automobile engineers with relation to the compression limits; in fact, it determines the limits of compression pressure which may be employed. It is well-known that benzene and ethyl alcohol will stand higher compressions without pre-ignition than will motor gasoline; the reason for this is to some extent explained when it is known that the spontaneous ignition temperatures in pure oxygen are as follows: benzene, 566°C. (1051°F.); ethyl alcohol, 395°C. (743°F.); and motor gasoline, 270° – 280°C. (518° – 536°F.), these temperatures being higher in air than in pure oxygen. Additional experimental work, however, is required before the method can be employed as a standard test.

(G) Composition.—The problem of the determination of the proportions of paraffins, olefines, aromatic hydrocarbons, and naphthenes in motor gasoline, as well as in all classes of petroleum oils, is of techno-chemical interest. This has been attacked with greater or less success by means of fractional distillation combined with fractional solution in liquid sulphur dioxide by Edeleanu's method,[1] and by S. E. Bowrey's modification of Edeleanu's method at lower temperatures,[2] while, among others, F. M. Perkin has investigated Valenta's proposal for the use of dimethyl sulphate as a fractional solvent for aromatic compounds and olefines.[3]

A further important paper on Edeleanu's method of separation as used for motor gasoline and kerosene has been published recently by R. J. Moore, J. C. Morrell and G. Egloff.[4] These authors used liquid sulphur dioxide at temperatures of −10°C. and −18°C. for treating known binary and ternary mixtures of pure toluene, benzene, p-xylene, mesitylene, hexylene, octylene,

[1] *Loc. cit.* (see page 34).

[2] *J. Inst. Pet. Tech.*, **3** (1917), No. 12, 287.

[3] *Ibid.*, 311. On the use of dimethyl sulphate, see Valenta, *Chem.-Ztg.*, **30** (1906), 266; Perkins and Harrison, *Analyst*, **33** (1908), 2; Reeves and Lewis, *J. Ind. Eng. Chem.*, **5** (1913), 293; Sommer, *ibid.*, **2** (1910), 5; and Church and Weiss, *ibid.*, **6** (1914), 396. Rittman, Twomey and Egloff (*Met. Chem. Eng.*, **13** (1915), 683) have reported that dimethyl sulphate is of no utility in the estimation of aromatic hydrocarbons in cracked petroleum.

[4] *Met. Chem. Eng.*, **18** (1918), 396.

cyclohexane, and other pure naphthenes, with pure light paraffins. They state that very good results can be obtained, provided the liquid sulphur dioxide used forms a sufficiently large proportion of the whole mixture, and the hydrocarbons in the mixture from which it is desired to separate one or more of them are also within certain specified limiting proportions. Contrary to the previously accepted view, they also find that naphthenes of low boiling-point can be separated completely from paraffins of low boiling-point if a sufficiently large volume of sulphur dioxide is used. It is worthy of note that in all these mixtures it is the denser constituent which is preferentially soluble in the sulphur dioxide.

(H) Vapor Pressure.[1]—The following method is the one prescribed by the Bureau of Explosives in connection with the shipment of gasoline (especially casinghead gasoline) in tank cars.

Apparatus (see Fig. 29).—Tube; pressure gage; water-bath for heating tube; and thermometer.

Procedure.—Remove the gage from the tube and fill the tube to 90 per cent. of its capacity. This is done preferably by lowering the tube into the storage tank in an upright position by means of a cord or wire. Leave the tube immersed for several minutes, then withdraw it, and pour off liquid until the tube contains 90 per cent. of its capacity. A small measure having a capacity of 10 per cent. of the tube should be used for this purpose.

In case it is impracticable to lower the tube into the storage tank, draw the liquid off into a vessel of a capacity about equal to that of the tube. Pour the liquid into the tube until it is about half-filled. Shake the tube and its contents gently in order to bring both to the same temperature. After standing for several minutes, pour out the liquid from the tube. Now

[1] Vapor pressure (or *tension*) determinations give direct information as to the probable behavior of motor fuels as regards easy starting and "flexibility," and therefore are considered by some automobile engineers in the evaluation of gasolines. Benzene and good quality gasolines possess high vapor pressures, and benzene also has the property of raising the ignition-point of mixtures. The explosive range is accorded attention in adjusting carbureting devices, so that the throttle scale may fall within the range, and the lower limit requires consideration in connection with vapor tension for fuels which are only explosive in mixtures containing comparatively high percentages. On the determination of vapor tension, see Philip, *J. Inst. Pet. Tech.*, **6** (1920), No. 21, 31; and Moore, *J. Soc. Chem. Ind.*, **39** (1920), 78T.

draw another sample from the storage tank into the cylinder and pour it through the funnel into the tube until the latter is entirely filled. Withdraw 1/10 as before. Screw the gage tightly into position.

Immerse the tube in water at 70°F. and allow it to remain for five minutes (the water should be stirred *constantly*). Remove the tube from the water and unscrew the gage sufficiently to relieve the pressure indicated by the gage for 20 seconds, then screw the gage tightly into place again. Place the tube in water at a temperature of 100°F. (90°F. from Nov. 1 to March 1). The level of the water must be just below the lower edge of the pressure gage. Stir the water continually and maintain the temperature exactly constant for ten minutes, then tap the gage lightly with the fingers and read the pressure.

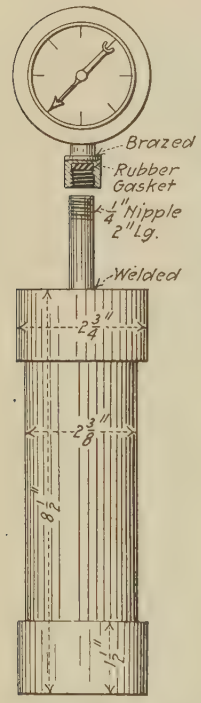

FIG. 29. Vapor pressure apparatus with gage connection used by the Atlantic Refining Company.

A correction of pressure figures should be made according to the initial temperature of the gasoline, as follows:

For tests on samples taken at a temperature of 59–50°F., deduct 1 pound.

For tests on samples taken at a temperature of 40–49°F., deduct 2 pounds.

For tests on samples taken at a temperature of below 40°F., deduct 3 pounds.

In making reports, the density of the liquid, the temperature of the liquid as placed in the tube, the pressure of the liquid at a temperature of 70°F. before venting the tube, and the corrected pressure at 100°F. (90°F. from Nov. 1 to March 1) after venting at 70°F., should be recorded.

5. Rules for the Shipment of Gasoline.—The Interstate Commerce Commission's regulations for the shipment of gasoline, are presented in the *Appendix*, page 334.

6. Gasoline Specifications.—Government specifications for gasoline, may be found in the *Appendix*, page 333.[1]

7. Results of Tests.—Reports in all cases should include the results of the following tests:

[1] See also DEAN, Bureau of Mines *Tech. Paper* **166.**

(1) Gravity in Baumé degrees at 60°F.

(2) Odor, color, acidity, water and dirt.

(3) Volatility. (A) Tabulated results of the distillation, which should include temperatures (observed and corrected) corresponding to each 10 c.c. of distillate collected, over-point, dry-point, and distillation loss. (B) Distillation curve plotted in ink on coördinate paper. (C) Barometric pressure. Temperatures should be reported in both Fahrenheit and Centigrade. Fahrenheit = 1.8 × Centigrade + 32.

In special cases the other tests, such as the "doctor" test, loss to sulphuric acid, etc., should be carried out.

II. PETROLEUM ETHER, SOLVENT GASOLINE, AND CLEANING OIL

Petroleum Ether.—The following description is taken from the *Pharmacopœia of the United States of America:*[1]

Benzinum Purificatum.[2]—A purified distillate from American petroleum consisting of hydrocarbons, chiefly of the marsh-gas series. Preserve it carefully in well-closed containers, in a cool place, remote from fire.

Purified petroleum benzine is a clear, colorless, non-fluorescent, volatile liquid, of an ethereal or faint petroleum-like odor, and having a neutral reaction. It is highly inflammable and its vapor, when mixed with air and ignited, explodes violently.

It is practically insoluble in water; freely soluble in ethyl alcohol, and miscible with ethyl ether, chloroform, benzene, volatile oils, and fixed oils, with the exception of castor oil. It has a specific gravity of 0.638 to 0.660 at 25°C. and distils completely between 40° (104°F.) and 80°C. (176°F.). Evaporate 10 mils (approximately 10 c.c.; see page 173) of purified petroleum benzine from a piece of clean filter paper; no greasy stain remains, and the odor is not disagreeable nor notably sulphuretted. Not more than 0.0015 g. of residue remains on evaporating 50 mils of purified petroleum benzine at a temperature not exceeding 40°C. Boil 10 mils of purified petroleum benzine for a few minutes with one-fourth its volume of an alcoholic solution of ammonia (1 in 10) and a few drops of silver nitrate T.S.; the liquid does not turn brown (pyrogenous products and sulphur compounds).

Add 5 drops of purified petroleum benzine to a mixture of 40 drops of sulphuric acid and 10 drops of nitric acid in a test-tube,

[1] Ninth revision, 74.

[2] Purified petroleum benzine, benzin. purif., or petroleum ether.

warm the liquid for about ten minutes, set it aside for half an hour, and dilute it in a shallow dish with water; no odor of nitrobenzene is evolved (benzene).

Solvent Gasoline.—A distillation curve for *solvent gasoline*[1] is presented in the *Appendix*, page 352. The product should be composed of "natural" hydrocarbons so refined that on distillation there is no decomposition with the formation of sulphur dioxide or of higher boiling polymerization products. It may be observed from the distillation curve that the product is fairly "close-cut;" that is, the initial and final boiling-points are comparatively close together.

Cleaning oil ("cleansing oil") is a "close-cut" naphtha product of boiling-point 120°C. (248°F.) to 150°C. (302°F.). It should combine the highest possible flash-point with the lowest final boiling-point consistent with that, and should possess a good odor and color. This product is used for dry-cleaning. Other grades of deodorized naphtha are marketed for use in paint, varnish and soap making. A distillation curve for naphtha is given in the *Appendix*, page 352.

Methods recommended for the examination of *turpentine substitutes* are as follows: Determination of color, gravity,[2] residue, and percentage of matter insoluble in sulphuric acid by the ordinary methods of turpentine analysis. Distillation may be carried out by the method given on page 343. The flash-point should not be below 85°F.[3] The evaporation test is made by placing 2 c.c. of the sample and the same amount of a standard substitute in watch-glasses in close proximity to one another and allowing the liquids to evaporate at room temperature. The sample examined should not require longer to evaporate completely than the standard substitute. In special cases 1,000 c.c.

[1] Solvent gasoline should not be confused with solvent naphtha, a coal-tar product. See discussion by Bacon and Hamor, *lib. cit.*, **2,** 887.

[2] "Turpentine substitutes" vary in gravity from 40° to 58° Bé. (usually 48° to 52°); and as they are intended for paint thinners and for admixture with turpentine, they should evaporate without leaving residues or stains (see Bacon and Hamor, *lib. cit.*, **2,** 918). On the use of benzine and other petroleum products as substitutes for turpentine in thinning paints, see Gardner's "Paint Researches," **1917,** 249; and *Circ.*, **94,** Educational Bureau, Sci. Sect., Paint Mfrs. Assoc. U. S., May, 1920.

[3] The U. S. Railroad Administration 1918 specifications for "mineral spirits" for paint-making require that the flash-point shall not be less than 85°F. (b.p., 265°F.–470°F.; 97 per cent. below 470°F.).

of the material under examination may be distilled until 100 c.c. remain. This residue then is distilled by a standard method to determine the properties of the high boiling 10 per cent. fraction of the sample. In thinning paints and varnishes, a very important point to consider in the selection of mineral spirits is that of solvent properties.

CHAPTER III

THE EXAMINATION OF ILLUMINATING OILS

1. Relative Importance of Tests.
2. Specific Gravity and Baumé Gravity.
3. Flash- and Burning-Points:
   Bureau of Mines Apparatus; Tagliabue Closed-Cup Apparatus; and New York State Board of Health Apparatus (Elliott) and Tagliabue Open-Cup Apparatus.
4. Sulphur.
5. Color.
6. Distillation.
7. Lamp-Test.
8. Other Tests Relating to Illuminating Oils:
   Acidity; Cloud-Point; Mineral Salts; and Viscosity.
9. Classification of Illuminating Oils.
10. Specifications for Illuminating Oils.

1. Relative Importance of Tests.—Ordinarily, the only tests necessary in the examination of illuminating oils are the determination of specific gravity, flash-point and color, and the lamp-test.[1] If the results of the lamp-test prove unsatisfactory, other tests may be applied for the purpose of definitely locating the trouble.

The determination of *odor* requires considerable experience, for it involves the exercise of judgment and consideration to the crude from which the illuminating oil is derived. It is necessary that the odor be sweet. In addition, no bad odor should result on burning the oil.

2. Specific Gravity and Baumé Gravity.—The specific gravity is determined by the methods described under gasoline (see page 41); the value obtained is changed into Baumé degrees at 60°F. (see the *Appendix*, page 312).

3. Flash- and Burning-Points.—The flash-point is the temperature at which an oil gives off vapor in sufficient volume to cause a flash (a burning of the explosive mixture of air and vapor)

[1] On the history of the stabilization of the manufacture and quality of kerosene, see CHANDLER, *Chem. Met. Eng.*, **22** (1920), 156 *et seq.*

when a flame is applied to the space above the oil under certain definite empirical conditions. The burning-point or fire-test is the temperature at which continuous burning takes place when a flame is applied to the space above the oil. These tests are important in connection with ascertaining the fire-hazard; in fact, the flash-test is quite essential since it is the real measure of inflammability.

The testers used for determining the flash- and burning-points of oils are either of the open- or closed-cup type, the former giving results which are higher than the latter because the vapor is not prevented from diffusing freely into the air. The apparatus, especially the open-cup tester, should be placed in a small box painted black on the inside (to facilitate the recognition of the flash or burning), and open on one side and on the top, when carrying out the test.

The rate of heating of ordinary lamp-oil having a fire-test up to 150°F. should be about 2°F. per minute. Mineral seal or other oils of 300°F. fire-test should be heated at the rate of 7° to 8°F. per minute.[1] Oils which contain water will have a higher flash-point than those free from that impurity. Before making a test for flash- or burning-point, the oil should be viewed in an oil-sample bottle, and, in case the oil is not clear, it should be treated to remove the dispersed water. For this purpose, filtration through dry filter-paper, centrifuging, or treatment with fused calcium chloride is employed.[2]

For accurate work, the observed temperatures (except when using special thermometers) should be corrected for the exposed portion of the thermometer stem by the method described under gasoline (see page 52) and for deviation from normal pressure according to the *Appendix*, page 348.

Allen and Crossfield have made a detailed study of the flash-points of oils and recommend the use of a modified Pensky-

[1] Tests on mineral seal, mineral colza, mineral sperm, 300° fire-test, and signal oils usually are taken in refineries with the Cleveland open-cup tester, the testing being started at 210°F. On this apparatus, see page 103.

[2] The electrical method of Cottrell also may be used. See Bureau of Mines *Tech. Paper* **49.** According to Philip (*J. Soc. Chem. Ind.*, **38** (1919), 394R), generally speaking, discrepancies in the observed flash-points are greater in the case of moist oils than in the case of dry ones. It is, however, unnecessary completely to dry oils having a flash-point of less than 200°F. before making tests.

Martens or Abel-Pensky apparatus.[1] Other forms of testers used widely are the Tagliabue closed-cup, the Tagliabue open-cup, the Foster and the Elliott. In case of dispute as to the true flash-point, the use of the Bureau of Mines apparatus is advised.

For a list of the State illuminating-oil inspection tests, see the *Appendix*, page 349.

The testers described herein for the determination of flash- and burning-points are as follows: (*A*) the Bureau of Mines modified Pensky-Martens and Abel-Pensky; (*B*) the Tagliabue closed-cup; and (*C*) the Elliott (New York State Board of Health) and the Tagliabue open-cup.

(A) Modified Pensky-Martens and Abel-Pensky Apparatus.—The Abel-Pensky apparatus, shown in Fig. 30, has a water-bath and is adapted especially to oils having low flash-points. The modified Pensky-Martens, shown also in Fig. 30, may be used successfully for almost any oil. The parts shown in Fig. 30 are as follows; 1, water-bath container; 2, jacket; 3, base-leg; 4, base-ring; 5, air-bath device; 6, jacket; 7, gauze-frame and gauze; 8, cup-lifter; 9, cover-lifter; 10, oil-cup; 11, oil-cup cover; 12, overflow-cover; 13, stirrer; 14, stirrer-shaft; 15, stirrer-sheave; 16, clock mechanism; and 17, thermometers.

Procedure for Flash-Point, according to Allen and Crossfield.—The approximate flash-point of an oil to be tested should be ascertained first by a preliminary test carried out by heating the sample so that the temperature will rise at the rate of 3°C. per minute, and applying the test after each 5° rise until the halo (see *infra*) appears and then at each 1° rise until the flash occurs. The test afterwards should be repeated carefully, in the following manner:

The sample and the oil-cup first must be brought to a temperature of at least 10°C. below the approximate flash-point of the oil, by standing the cup in an ice mixture, if necessary, before the cup is adjusted in the bath. The lower edge of the overflow aperture is greased on its outer side to induce ready overflow when the oil expands. The clean, dry cup then is placed in the bath, and the sample is run into the cup with the aid of a glass pip-

[1] Bureau of Mines *Tech. Paper* 49. For a review of the literature on flash-point determination and explosion hazards of kerosene, see Robson and Withrow, *Oil, Paint and Drug Reporter*, 96 (1919), No. 4, 30, and No. 5, 17.

ette until the filling-point just disappears under the surface, as seen by light reflected from the surface of the oil. Care must be taken not to splash the oil to the sides of the cup and not to have froth formed on the oil. All bubbles on the surface of the oil must be punctured with a heated wire. In case too much oil has been run accidentally into the cup, the cup must be emptied, washed clean with a good solvent (*e.g.*, naphtha), wiped dry, and a fresh filling made. After the filling has been done correctly, adjust the cover and thermometer immediately, light the test

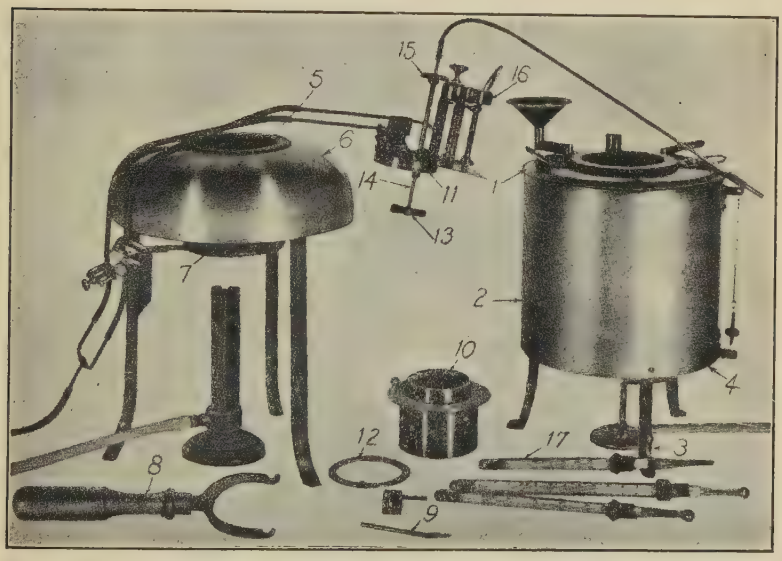

FIG. 30.—Bureau of Mines modified Pensky-Martens (at left) and Abel-Pensky (at right) testers dismantled to show parts.

flame, and regulate it to the size of the ivory bead on the cover—that is, so that it will burn 0.1 cu. ft. of coal gas per hour.

Allow the apparatus to stand 10 minutes, to give time for the oil vapors to accumulate, meanwhile stirring regularly and constantly at one revolution per second. Then light the gas flame below the bath and adjust it to such a height, as determined by preliminary tests, that the temperature of the oil will rise at the rate of about 2°C. per minute. Warm to within 5°C. of the predetermined flash-point and expose the test flame for exactly one second by means of the mechanism provided on the cover. Continue stirring and making the exposure at each 0.5°C. rise

in the temperature until the flash occurs. Particular care must be taken that the cup is not subjected to drafts during the test and that the breath of the operator is not allowed to interfere, particularly at the moment the test flame is exposed to the vapors. It will be noted that, when the oil is about 5°C. below the flash-point, the test flame, as it is exposed to the vapor, will be surrounded by pale-blue halo, which gradually increases in intensity until a sudden inflammation, or gentle explosion, of the vapors, or the "flash" occurs. The temperature at which this takes place, as registered by the thermometer in the oil, is the flash-point. With fuel-oil residues or with poorly refined oils from which a small quantity of low-flashing fractions is being liberated continually, the described halo may appear first at a temperature as much as 30°C. below the flash-point.

The test always should be repeated with a fresh sample. The first sample should be thrown away, the cup washed with naphtha, wiped dry, the cup and water-bath cooled to the proper temperature, and the test repeated exactly as described above. Duplicate tests should agree within 0.5°C. The mean of the two readings, if good agreement is noted, corrected for barometric pressure, is considered the flash-point of the oil.

Burning-Point.—The burning-point is that temperature at which the oil ignites and continues to burn. To determine the burning-point, open the cover, continue the heating, and expose the test-flame to the vapors in the same manner as when taking the flash-point, until the oil inflames and burns continuously.

(B) The Tagliabue Closed-Cup.—See Fig. 31,[1] wherein A is the thermometer showing the temperature of the oil; B, the thermometer indicating the temperature of the water-bath; C, the oil-supply chamber for the test flame when gas is not available, mounted on the axis about which the test flame is rotated, which axis is hollow and provided with a connection on the end for the gas hose and also with a needle-valve for controlling the gas supply when gas is available, the gas passing through the empty oil chamber; D, the gas or oil tip for the test flame; E, the cover for the oil-cup, provided with three openings, which are, in turn, covered by a movable slide operated by a knurled hand-knob, which also operates the test flame burner in unison with the movable slide, so that, by turning the knob, the test flame is lowered into the middle opening in the cover at the same time

[1] American Society for Testing Materials standard for turpentine substitute (*Proc.*, **18** (1918), 1, 689).

that this opening is uncovered by the movement of the slide; F, the oil-cup; G, the overflow spout; H, the water-bath; J, the body; K, an alcohol lamp; and L, gas hose.

Procedure.—If gas is available, a ⅛-in. rubber tube is attached to the gas connection on the oil-cup cover. If no gas is accessible, the test flame burner tip is unscrewed from the oil chamber and a wick of cotton cord is inserted in the burner tip, which is replaced. A small quantity of cotton waste and some signal, sperm or lard oil are placed in the chamber, the wick is lighted and the test flame adjusted to the size of the small white bead mounted on the top of the apparatus.

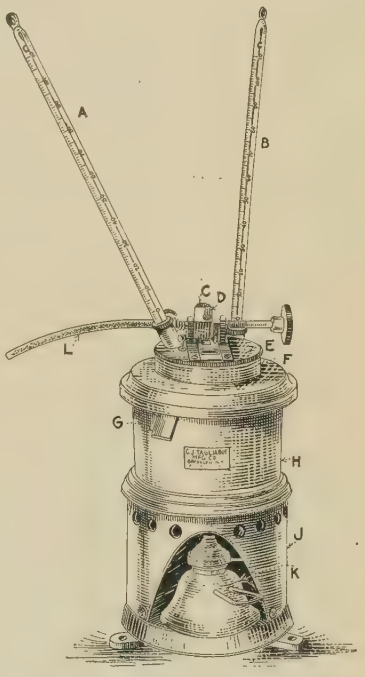

FIG. 31.—The "Tag" closed tester for the determination of flash-point.

The apparatus should be placed firm and level, and shielded from draughts on three sides. Insert the bath thermometer and fill the water-bath with water at a temperature at least 10°C. below the probable flash-point of the oil to be tested. A vessel should be placed underneath the overflow spout to receive the excess water from the bath.

Next insert the oil-cup in place, measure 50 c.c. of the oil to be tested and place it in the cup. Destroy any bubbles which may be on the surface of the oil. Adjust the cover in place with the thermometers and gas tube attached, ignite the pilot light and regulate the flame to the size of the small white bead on the cover.

Observe and record barometric pressure, temperature of water-bath and temperature of oil.

Light the heating lamp and locate it centrally at the base of the apparatus. Regulate the flame of the lamp so that the temperature of the oil rises at the rate of 1°C. per minute (not faster than 1.1°C. nor slower than 0.9°C.).

When the temperature of the oil reaches about 5°C. below the probable flash-point of the oil, turn the knob on the cover so as to introduce the test flame into the cup and turn it promptly back again. Do not permit it to snap back. The time consumed in turning the knob down and back should be about one full second, or the time required to pronounce distinctly the words "one thousand and one."

Observe and record the time of making the first introduction of the test flame and the temperature of the oil at the time of the first test.

Repeat the application of the test flame for each 0.5°C. rise in temperature of the oil until there is a flash within the cup. Do not be misled by the halo about the test flame or by the slight flickering of the flame; the true flash consumes the vapor in the top of the cup and causes a very slight explosion.

Record the time and temperature at which the flash occurs. The latter is the flash-point.

Fig. 32.—The New York State Board of Health (Elliott) apparatus.

Repeat Tests.—Having completed the preliminary test, lift up the oil-cup cover and wipe off the thermometer bulb. Lift out the oil-cup, remove the oil, and wipe out the cup carefully.[1] Pour water into the water-bath, permitting it to overflow until the temperature is at least 8°C. below the flash-point of the oil.

Place the oil-cup back in the bath and measure into it 50 c.c. of the fresh oil. Destroy any bubbles on the surface of the oil, adjust the cover in place, and proceed to make a second test.

The experiments should be repeated until checks are had to within 0.5°C. Finally, the observed flash-point is corrected to standard pressure by the use of the *Appendix*, page 348.

(C) New York State Board of Health Apparatus and the Tagliabue Open-Cup Apparatus.—The first-mentionedis shown in Fig. 32. The cup is covered by a glass plate having two holes, one for the thermometer and the other for inserting the test flame. In making the test, it is specified that the test flame be inserted into the hole in the cover to a distance one-half

[1] It may be advantageous to rinse the cup with a solvent such as ethyl ether, and then to dry carefully.

way between the cover and the oil, with "a motion steady and uniform, rapid, without any pause." In filling this apparatus, enough oil is poured in to fill the cup to within $\frac{1}{8}$-in. of the flange separating the cup and vapor-chamber above.

The Tagliabue open-cup apparatus (see Fig. 33) has a glass cup, above which is fastened a guide wire along which the test flame is passed. The oil cup is filled to within $\frac{1}{8}$-in. of the upper level edge. In using any form of open-cup apparatus especial care should be taken that draughts are eliminated, and that the flash spreads completely across the surface of the oil. A partial flash, originating at the edge, is not the true flash.

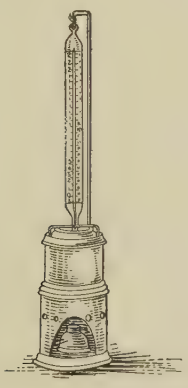

FIG. 33.—The Tagliabue open-cup apparatus.

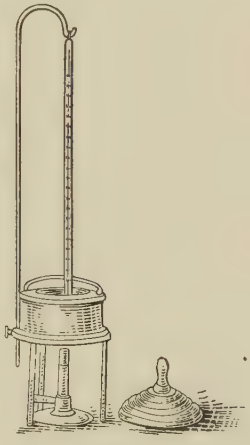

FIG. 34.—The Cleveland open-cup tester, used principally in the examination of lubricating oils (see also Fig. 45).

4. Sulphur.—Sulphur in illuminating oil is a matter of considerable importance since sulphur compounds occurring in the oil may produce a disagreeable odor during its burning, while the oxidation products of the impurity may cause carbonization of the wick. The amount of sulphur in good illuminating oils should not exceed 0.06 per cent., but many lower grades contain as much as 0.10 per cent.

In determining the percentage of sulphur in illuminating oils, some form of lamp apparatus generally is used because of the fact that, by this method, larger samples may be treated; then, too, the bomb method is much more tedious and the lamp method is sufficiently accurate. An apparatus somewhat similar to that

shown in Fig. 28 may be fabricated from ordinary laboratory equipment. The lamp may be made from an Erlenmeyer flask, a cork, a glass tube for holding the wick, and a suitable wick. The absorption apparatus may contain short lengths of hard glass tubing. The procedure is to place 5 or 6 c.c. of the oil in the Erlenmeyer flask and then to weigh the whole lamp to the third place. A current of air is drawn through the apparatus, the lamp is placed under the hood, and the wick is lighted. Previously 5 c.c. of N/10 sodium carbonate solution have been placed in the absorption apparatus, where it will come into intimate contact with the products of combustion. The operation is continued for about 30 minutes, when the flame is extinguished and the lamp is weighed. Finally, the sodium carbonate solution is removed and the absorption apparatus is washed with distilled water neutral to an indicator. The combined solutions then may be titrated with N/10 sulphuric acid (1 c.c. of which is equal to 0.0016 g. of sulphur), using methyl orange as the indicator; but preferably are oxidized with bromine and hydrochloric acid, and the sulphuric acid precipitated with barium chloride solution in the usual way and calculated to sulphur.

Notes and Precautions.—A useful form of lamp and accessory apparatus has been described by Heusler and Engler.[1] By this method the sulphur dioxide produced by burning the oil is oxidized to sulphur trioxide and absorbed by a solution of sodium hypobromite. The sulphate finally is precipitated by barium chloride solution in the customary manner. The lamp also is arranged so that air from the outside may be drawn through a rubber tube to the hood above the lamp. This procedure eliminates the danger of drawing contaminated laboratory air into the apparatus.

The lamp method fails to estimate sulphur present as sulphuric acid or derivatives of sulphuric acid, unless the wick is afterward digested with fuming nitric acid and the sulphur determined.[2] Some types of sulphur compounds are difficult to oxidize,[3] and, if they are present, more efficient methods of oxidation must be used. An 18-in. silica tube packed with spongy platinum and heated red hot, may be interposed between the lamp and the absorption apparatus.

[1] *Chem.-Ztg.*, **20,** 197.

[2] See page 97.

[3] Thiophenes, for example.

The Determination of Sulphur According to Conradson.—In the examination of the illuminating (kerosene) oils used in railroad service, such as in locomotive headlights or automatic block signal lamps (long-time burner lamps), which latter require an oil with hardly any diminution in the intensity of light or candle power for 150 to 250 hours' continuous burning with no attention; or lubricating oils, such as used in steam turbines, gas or oil engines, superheated steam valve and cylinder lubrication, high

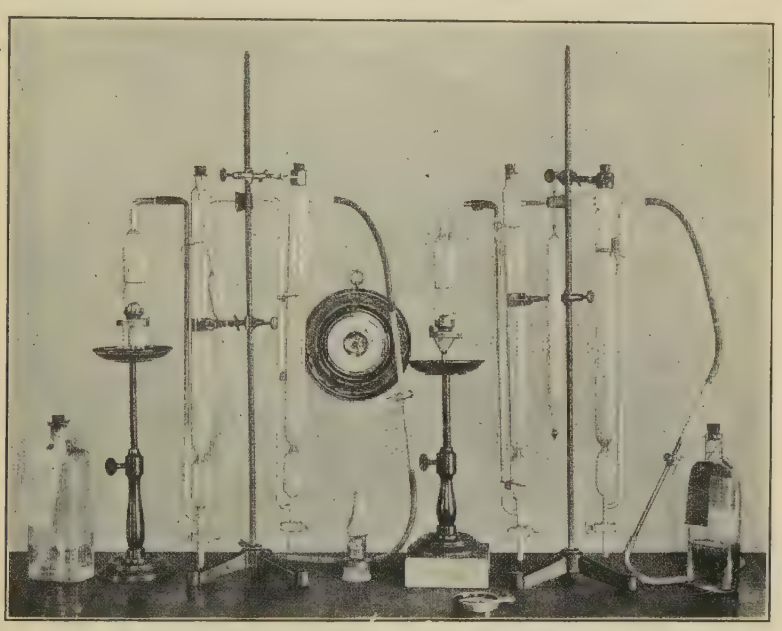

FIG. 35.—The apparatus used in Conradson's method for the determination of sulphur in illuminating and lubricating oils.

pressure air compressors or high vacuum air pumps, it is essential to differentiate between the sulphur compounds that might be present in the oils. Therefore, Conradson[1] has found it necessary not only to burn a larger amount of oil in the lamps, but also to consume all the oil in the oil fount and make a careful examination of the sulphur compounds that may remain in the wick from the oil.

Fig. 35, which shows Conradson's apparatus, is self-explanatory—using ordinary small kerosene burners with chimneys well

[1] *J. Ind. Eng. Chem.*, **4** (1912), 842. See also page 97.

washed and dried cotton wicks about 3 mm. (⅛ in.) in width. The lamp founts for the illuminating oils are ordinary glass beakers; for the lubricating oils, funnels having the stems cut off and fastened to a metal socket. The burners are inserted in a small disk (lid of an ointment box with a hole cut to fit the burner). Filter tubes of strong glass with stem bent at right angles are fit snugly over the long arm of the glass stoppered absorption tubes, which are about 350 mm. (14 in.) in length between the constriction and exit tube and have a diameter of about 25–35 mm. (1 in.–1¼ in.) and contain small glass beads to a depth of about 80 mm. (3 in.).

The products of combustion are aspirated through sodium carbonate solution containing 6 g. of sodium carbonate in 1 liter of water and standardized with N/10 hydrochloric or sulphuric acid. In testing, place 25–50 c.c. of the soda solution in the first absorption tube. At the end of the operation the soda solution is run into a beaker and the chimneys, glass filter tubes and absorption tubes are rinsed out with water; the solution with the washings is titrated, using methyl orange as an indicator; or the solution may be oxidized with bromine and hydrochloric acid, precipitated with barium chloride solution in the customary manner and calculated to sulphur. The wicks are treated separately and examined for sulphur compounds that may remain from the oil.

For low-sulphur oils, such as kerosene made from Pennsylvanian crudes, 15–20 g. of oil are used.

For kerosenes made from western crudes containing larger percentages of sulphur compounds, 10–15 g. are used.

For kerosene oils intended for severe railroad service, either for headlight oil purposes or in long-time burner signal lamps, it is well to make two sulphur determinations, one as described and the other as follows: 425 c.c. of the oil are distilled carefully in a 600-c.c. Engler distilling flask at the rate of 2–4 c.c. per minute (the slower rate at the beginning and at the end), until 400 c.c. have come over; the Engler flask is placed on an asbestos gauze and covered over with asbestos wool up to the top of the neck, the thermometer bulb placed opposite the exit tube as usual. The 25 c.c. residue in the distilling flask is transferred to a bottle. Often with ordinary or poorly refined kerosene oils there is formed a deposit or sediment, more or less adherent to the sides and bottom of the flask; it should be removed

carefully and added to the residue in the bottle by using successively ethyl ether, chloroform, 95 per cent. ethyl alcohol and hot water (as the case may require), uniting and evaporating the solvents in a small dish and transferring the residue to the bottle containing the 25 c.c. residue. The sulphur compounds then are determined as explained *supra*.

The "Doctor" Test.—This test, as described under gasoline (see page 59), applies also to illuminating oils.

5. Color.—The practice of determining the color of an illuminating oil by comparison of a 4-oz. sample in a flat bottom bottle with a standard, does not admit of accurate results and has given way to the use of chromometric methods. The standard instruments are those of Wilson, Stammer and Saybolt.

The color of illuminating oil varies from that known as *standard white* (zero Saybolt; 50 mm. Stammer) through the grades known as *urime white* (86.5 mm. Stammer) and *superfine white* (199.5 mm. Stammer) to *water-white* (21 Saybolt; 300–320 mm. Stammer). A comparison of Saybolt and Lovibond values with equivalent solutions of potassium dichromate is given in the *Appendix*, page 353.

(A) Wilson and Stammer Chromometers.[1]—The instrument of *Wilson* consists of two nearly upright tubes having glass caps at their bottoms. A mirror reflects light from below through the two tubes into the eyepiece at the top. In ascertaining the color, one of the tubes is filled with the oil, while the other is left empty and beneath it the standard color glasses (good merchantable, standard white, prime white, superfine white, and water-white) are placed, successively, until the closest possible agreement is had between the color of the oil and that of one of the glasses. The *Stammer* instrument has two vertical tubes, one shorter than the other. The standard color glass is inserted at the top of the longer tube. The oil to be examined is poured into a cylinder having a colorless glass bottom and is placed beneath the shorter of the two tubes. By means of a wheel the height of the column of oil observed through the eyepiece may be varied by lowering or raising the cylinder until the color of the oil agrees with that of the color glass. The color is calculated from the length of oil column observed, in millimeters.

(B) Saybolt Universal Chromometer.—This instrument, which is the most popular in the United States and admits of

[1] For detailed descriptions of these instruments, see Redwood's "A Treatise on Petroleum," **2**, 214.

very accurate tests (see Fig. 36), consists of two removable glass cylinders (color comparison tubes), one graduated. The oil is contained in the graduated tube, while the standard color glass is inserted at the bottom of the other. Two like-colored yellow glass discs are supplied with each chromometer: by the use of one singly or both together,[1] color shades can be determined from below 0 to +25.

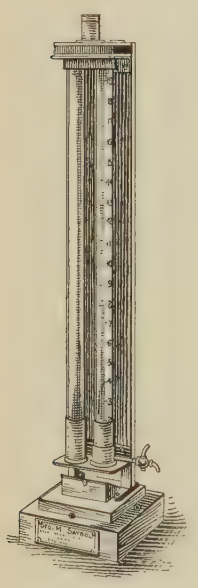

FIG. 36. –The Saybolt chromometer, for the determination of the color of illuminating oil and gasoline.

Procedure.—The oil is placed in the graduated tube, and the color glass or glasses are inserted at the lower end of the other tube. By means of the valve at the bottom of the graduated tube, the oil is withdrawn until the color observed through the eye-piece at the top agrees with that of the standard glass or glasses. The color then is expressed by the height of oil column (3 to 20 in.) in scale divisions, the higher the reading the better the color. Four and four-eighths in. of oil in the tube, using two discs, is equivalent to 0 color shade, or standard white; and 12 in. of oil in the tube, using one disc, equals + 21 color shade, or water white.[2] When the tube filled with oil is lighter in color than the comparison tube with only one disc, the color is read + 25 and up (better than + 25).[3]

6. Distillation.—Data secured by the distillation of illuminating oils may give the cause for the decrease of the illuminating power of the oil after burning for a time. The apparatus used is the Engler flask and the rate of distillation is 2½ c.c. per minute. According to Lockhart[4], oils which have more than 5 per cent. by weight of residue above 300°C. (572°F.) are likely to give poor results by the lamp test. The boiling-points of the best lamp-oils lie between 150° C. (302°F.) and 300°C. (572°F.). This does not

[1] The two glasses are used to determine color shades up to and including +15, and only one glass from +16 to +25.

[2] 21 color is equivalent to 0.0048 g. of potassium dichromate per liter of water.

[3] For a table of color shades, see the *Appendix*, page 354.

[4] "American Lubricants," **1918,** 215.

apply to such oils as mineral seal, which has a flash-point around 250°F. and an initial boiling-point of 250° to 260°C. (482°–500°F.).

7. Lamp or Burning Test.—Good illuminating oils are composed of hydrocarbons of the paraffin and naphthene series, the other hydrocarbons and impurities, such as sulphur compounds, having been removed, substantially, during the refining processes. Aromatics and olefines give a smoky flame, while sulphur compounds, such as alkyl sulphides and derivatives of thiophene, cause a disagreeable odor during burning. Derivatives of sulphuric acid cause charring of the wick and mineral (usually sodium) salts produce incrustation of the wick. If the kerosene is not sufficiently "close cut," that is, if there is much of the high boiling fraction (above 300°C.) present, the flame becomes lowered after a time.[1]

In order that an illuminating oil may burn normally, it is essential that the wick shall furnish sufficient oil to the flame. The rise of the kerosene in the capillaries is determined by the surface tension that the oil exerts therein, while the viscosity of the oil opposes the rise by increasing the resistance in the capillaries.

The flow of burning oil through a wick has been investigated by Stepanow.[2] Mathematically, the results obtained are expressed by the equation:

$$Q = A\frac{\alpha^2}{Z}.$$

Q is the volume of oil drawn to a certain height through the wick, α the capillary constant of the oil, and Z the viscosity of the oil. A is a rather complicated factor, involving the specific gravity of the oil. For a given wick, however, under fixed conditions, no great error is involved in regarding A as a constant. Since the upper portion of the wick is heated by the burner, the changes in viscosity and capillary constant of an oil with temperature are of importance. Both decrease, the former, however, much more rapidly than the latter. This behavior is of value since it tends to maintain the height of the flame as the oil level falls in the

[1] The presence of paraffin in kerosenes is said, however, to have very little effect on burning quality (see NEWTON and WILLIAMS, *Petroleum Age*, **6** (1919), No. 3, 83).

[2] "Grundlage der Lampentheorie," **1896.**

lamp reservoir. For very viscous oils, the burner is lengthened so that it dips into the oil, in order to raise its temperature.

The ultimate test of an illuminating oil is actual burning in a lamp for a time and noting the behavior. This conclusively indicates the quality of an oil—that it will burn, how it burns, and how much oil is consumed in a given period of time.

Burning Test.[1] *Water-White Kerosene.*—The oil must burn freely and steadily in a lamp fitted with a No. 1 sun-hinge burner. It must give a good flame for a period of 18 hours, without smoking or forming "ears" or "toadstools" on the wick. The chimney must be only slightly clouded or stained at the end of the test.

Long-Time Burning Oil.—The burning test is to be made by introducing 25 fluid ounces of oil into the pot of a standard Railway Signal Association semaphore lamp, fitted with the purchaser's standard burner, chimney and wick. The wick shall be new and previously washed with redistilled ethyl ether and dried at room temperature; the lamp is to be protected from the direct rays of the sun, but may be burned either outdoors or in a well ventilated room. During the first hour of the test, the wick shall be adjusted so as to produce a flame seven-eights of an inch high, measured from the top of the wick. The lamp shall burn continuously for 120 hours, without readjusting the wick, or until all the oil is consumed.

300-*Degree Mineral Seal.*—The burning test is to be made by introducing 20 fluid ounces of the oil into a lamp fitted with a dual burner No. 3, dual chimney and duplex wicks. The lamp used shall be such that the distance from the top of the wick tube to the bottom of the inside of the fount is not less than six and one-half nor more than seven inches. During the first hour of the test the wicks shall be adjusted so as to produce a symmetrical flame, approximately one inch high, measured from the top of the wicks. The lamp shall burn continuously without readjustment until all the oil is consumed. The flame shall remain symmetrical and free from smoke throughout the test period. The oil shall not produce any appreciable hard incrustation on the wick.

Signal Oil.—Signal oil is tested in a standard railway lantern (see Fig. 37) under practical conditions. An observation must be made of the length of time the lantern will burn with an effective flame without trimming the wick, and it is also essential to

[1] Method of the United States Fuel Administration.

determine whether the lantern will burn without smoking, regardless of the angle at which it is held or swung.

8. Other Tests Relating to Illuminating Oils.—The presence of *free acid* in kerosene may be determined by the method described under gasoline (see page 41). Illuminating oils should show neither an acid nor an alkaline reaction.

The *cloud-point* of ordinary kerosene is generally around 0°F. and many specifications call for a cloud-point not above that temperature. The chill or cloud test indicates to what extent paraffin is present.

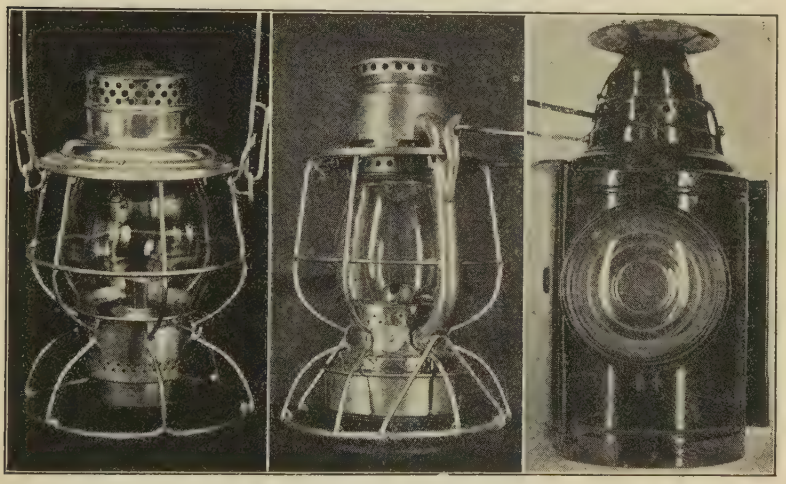

FIG. 37.—Two types of trainmen's hand-lanterns and a signal semaphore lamp. (Reading from left to right: Pennsylvania Railroad trainmen's lantern; trainmen's kerosene-burning hand-lantern; and signal semaphore lamp.)

CLOUD TEST.[1] *Water-White Kerosene.*—Take a four-ounce sample bottle and introduce therein one and one-half ounces of the oil to be tested; insert the cork with a cold-test thermometer, so that the thermometer is suspended in the oil. Place the bottle in a freezing-mixture and cool to 0°F. Keep the oil at that temperature for ten minutes. The bottle should be given a rotary motion occasionally, so as not to supercool the sides. The oil should not be clouded from crystals of paraffin at the end of ten minutes.

Long-Time Burning Oil.—Same as for *water-white kerosene.*

[1]Method of the United States Fuel Administration.

300-*Degree Mineral Seal.*—Same as for *water-white kerosene*, except that the oil is cooled to 32°F.

The chill test of *signal oils* will depend not only upon their source as to crude, but also upon the content of fatty oil. The chill varies from 10° to 50°F.

The Flock-Test[1] also is called for in various specifications. It is applied as follows:

Water-White Kerosene.—Take a hemispherical iron dish, and place a small layer of sand in the bottom. Take a 500-c.c. Florence or Erlenmeyer flask and put into it 300 c.c. of the oil (after filtering, if it contains suspended matter.) Suspend a thermometer in the oil by means of a cork slotted on the side. Place the flask containing the oil in a sand-bath, and heat the bath so that the oil has reached a temperature of 240°F. at the end of an hour. Hold the oil at a temperature of not less than 240°F. nor more than 250°F. for six hours. The oil often becomes discolored, and this may be expected, but there should be no suspended matter formed therein. The flask should be given a slight rotary motion; and, if there is a trace of flock, it can be seen to rise from the center of the bottom.

Long-Time Burning Oil.—Same as for *water-white kerosene.*

300-*Degree Mineral Seal.*—Same as for *water-white kerosene*, except that the heating is at 10°F. per minute to a temperature of 450°F. and is held at that temperature for fifteen minutes. The oil should show no flock or precipitate at that temperature, or in one hour after cooling.

Mineral salts may be determined by distilling 500 c.c. of the oil to small bulk, evaporating to dryness, igniting, and examining the residue qualitatively and quantitatively for mineral constituents. The ash content of properly refined illuminating oils will be very low and in most cases practically *nil.*

The *viscosity* of illuminating oil is determined by means of a viscosimeter having a jet of smaller diameter than those of the instruments used for lubricating oils. In this way the Engler instrument has been modified by Ubbelohde for use in determining the viscosity[2] of illuminating oils and naphtha products

[1] Method of the United States Fuel Administration.

[2] This apparatus is described in HOLDE-MUELLER'S "Examination of Hydrocarbon Oils," **1915,** 56. Cf., however, HERSCHEL, *Proc. Am. Soc. Testing Materials*, **17** (1917), II, 551; and *Tech. Paper* **125,** Bureau of Standards.

(spirits).[1] The Saybolt thermo-viscosimeter also may be employed. The theoretical relations among flow of oil through the wick, capillary constant, and viscosity,[2] are discussed on page 81.

Illuminating oils should be clear and bright and free from suspended matter. The suspended matter may be water, glue, dirt, or a combination of the three. To test for suspended matter,

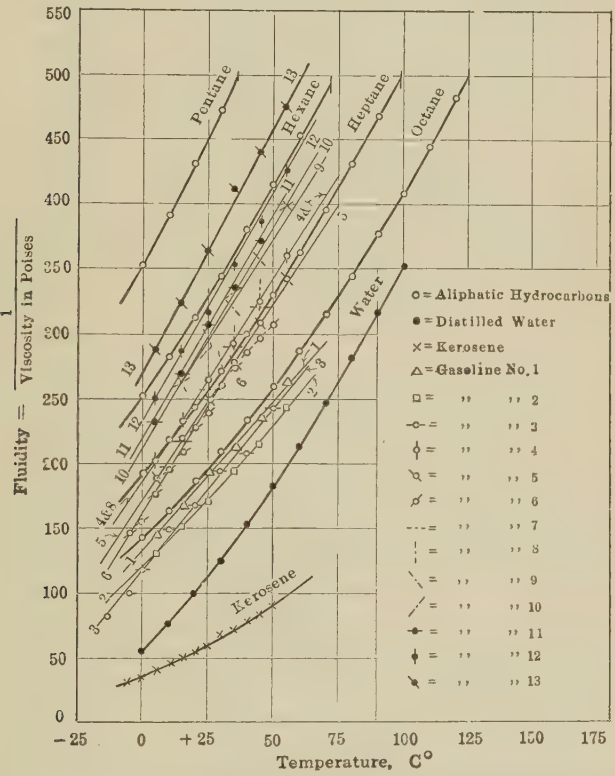

FIG. 38.—Temperature-fluidity curves of aliphatic hydrocarbons, kerosene, and gasoline.

the oil is viewed in an ordinary oil-sample bottle. As mentioned, the odor of kerosenes should be characteristically sweet.

[1] According to HERSCHEL (*supra*), the Ubbelohde viscosimeter serves well enough for a commercial determination of the fluidity of gasoline.

[2] GEORGE H. PERKINS, of Philadelphia, Pa., was the first to realize that the viscosity of an illuminating oil was a measure of its wick-climbing ability and consequently of its burning performance. He devised, sometime in the '80's, accurately made, special forms of viscosimeters for determining viscosities.

9. Classification of Illuminating Oils. *Ordinary Lamp-Oils.*—These products have a Baumé gravity of from 40° to 49°, a flash-point varying from 110° to 130°F., and a fire-test of from 125° to 160°F. *Export kerosene* ("export oil") is usually of 44° Bé. gravity, or lighter; that for England is of 73° Abel flash-point and that for Germany has a flash-point of 21° Celsius.

Mineral Seal or Mineral Colza.—A special oil having a flash-point around 250°F. and fire-test around 300°F., largely used in lighting railway coaches.

Signal oils are made by compounding fatty oils with mineral seal.[1]

Long-Time Burning Oil.—The specifications for this product correspond to those for ordinary illuminating oil, except that a longer burning test is required.

10. Specifications for Illuminating Oils.—General specifications for illuminating oils may be found in the *Appendix*, page 488.

[1] The percentages used vary considerably and various fatty oils (sperm, peanut, and prime lard) are employed by different refiners. The fatty oil used should be neutral or nearly so. On sperm oils used for burning purposes, see BLAKELEY and REILLY, *J. Ind. Eng. Chem.*, **9** (1917), 1099.

The signal oil used on the Lines East of the Pennsylvania Railroad Co. consists of a mixture of 30 parts by volume of prime lard oil and 70 parts by volume of 300° mineral seal oil; the compounded oil complies with the requirements of Specification No. **R914** of the United States Railroad Administration.

CHAPTER IV

THE EXAMINATION OF LUBRICATING OILS AND GREASES

1. Relative Importance of Tests Concerning Lubricating Oils and Greases.
2. Specific Gravity and Baumé Gravity.
3. Color.
4. Viscosity.
5. Free Acid.
6. Sulphur and Organic Sulphur Compounds.
7. Saponification Number.
8. Soaps.
9. Cold Tests.
10. Flash and Fire Tests.
11. Carbonization.
12. Volatility.
13. Emulsification.
14. Maumené Number.
15. Special Tests on Valve and Cylinder Oils.
16. Lubricating Greases.
17. The Theory of Lubrication and the Coefficient of Friction.
18. Specifications for Lubricating Oils.
19. Blending of Lubricating Oils.

1. Relative Importance of Tests Concerning Lubricating Oils and Greases.—It will be found of advantage to examine all lubricating oils for specific gravity, color, viscosity, free acid, flash-point and cold test. When comparing oils, the heat and volatility tests also may be included. Additional tests may be applied to oils for special purposes, such as the determination of the demulsibility of oils used in forced-feed lubrication, the determination of the carbonization value for automobile cylinder and air compressor oils, and the saponification number of and perhaps the identity of the fatty oil used in compounded cylinder oils. Odor sometimes assists in the detection of the kind of fatty oils in compounded oils. In the case of greases it is, of course, essential to know the melting-point, and, in addition,

the product may be analyzed to identify and determine the percentage of the constituents in the mixture.[1]

General requirements for lubricating oils and greases for various purposes are presented in the *Appendix*, page 380. For the methods of testing lubricating oils for Government purchase, see the *Appendix*, page 558.

2. Specific Gravity and Baumé Gravity.—The determination of specific gravity renders the chemist able in many cases to form an opinion respecting the nature of the crude petroleum from which the lubricating oil has originated, as an aid in duplicating it.

Specific gravity may be found accurately with the pycnometer[2] shown in Fig. 20. The observation is taken with the sample at 60°F. (15.56°C.), compared with water at the same temperature, and the result is reported in the Baumé scale equivalent. For the more fluid oils the hydrometer or the Westphal balance may be used, but these instruments always should be verified. Correction for the buoyant effect of the atmosphere should be made when necessary.

3. Color.—The color, along with the degree of transparency, consistency and odor, affords the experienced chemist an indication as to the source of the lubricating oil under examination and provides hints for selecting the best methods of evaluation. Then, too, *ceteris paribus*, an oil with a lighter color or pleasing appearance generally is preferred to a darker colored oil. Lubricating oils should be free from water, suspended matter[3] and turbidity.

Therefore the color always should be observed and the Saybolt chromometer (see page 80) may be used satisfactorily in the examination of light-colored lubricating oils. However, the employment of the Lovibond tintometer (Fig. 39) generally is preferred for the determination. The Lovibond instrument

[1] Conradson has discussed the interpretation of analyses of lubricants in *J. Ind. Eng. Chem.*, **2** (1910), 171. See Everest, *Proc. Eng. Congress*, Chicago World's Fair, **1893,** for an earlier paper on the same subject.

[2] The determination of the specific gravity of high viscosity oils may be carried out conveniently in the Hubbard pycnometer (*J. Ind. Eng. Chem.*, **1** (1909), 475). See page 127.

[3] Dark-colored oils, heavy machine oils and cylinder oils sometimes are found to contain carbonaceous matter in suspension upon examining several drops on a slide under the microscope. Paraffin crystals, if present at room temperature, will disappear upon warming the oil, and other foreign matter held in suspension will be brought out.

is divided longitudinally by a partition, and the sample to be tested, contained in a cell of suitable length, is placed on one side and matched by the standardized color glasses inserted in the slot on the other side. In carrying out the determination it is advised that a piece of white paper be pasted over the window and

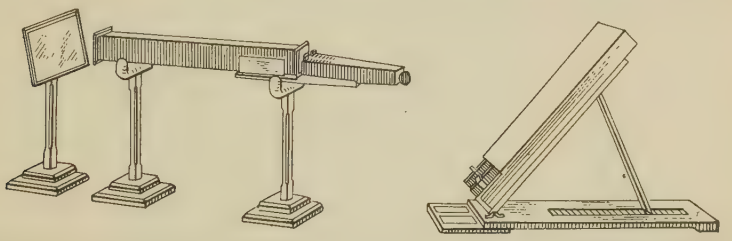

FIG. 39.—The Lovibond tintometer (*J. Soc. Dyers and Col.*, **1887**, 186; **1894**, 3, 22 and 206).

the instrument directed against the paper. The cells used are of varying length, depending upon the depth of color of the sample under examination. While results of accuracy cannot be obtained by the use of tintometric methods, comparative results on oils of the same shade are thus found.[1]

The determination of color according to the standards of the American Petroleum Association may be carried out by means of the Union colorimeter (see Fig. 40), as follows:

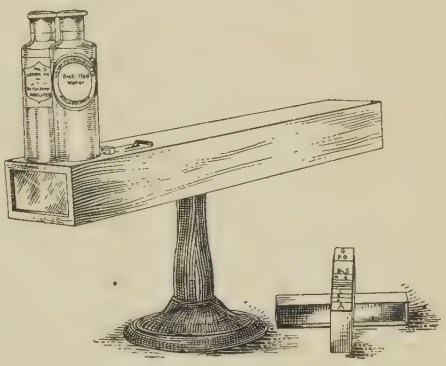

FIG. 40.—The Union colorimeter.

For lubricating oils other than cylinder oils the oil is placed in a four-ounce sample bottle and then inserted in one of the circular compartments of the instrument. In the opposite compartment is placed a four-ounce bottle filled with distilled water. One of the standard glasses then is placed in the slot and the slide is closed. The instrument finally is taken to a window, where the observer, looking through, can compare the color of the oil with the standard glass.

[1] On the Lovibond tintometer and its application to petroleum products, see CAMPBELL'S "Petroleum Refining," **1918**, 74 *et seq.*

Cylinder oils are mixed first with water-white gasoline, in the proportion of 15 per cent. of oil and 85 per cent. of gasoline, and the determination is proceeded with as in the case of other lubricating oils.

4. Viscosity.—Viscosity may be defined as the internal friction of fluids. The absolute viscosity of an oil is the tangential force in dynes necessary to move a unit area of plane surface with unit speed relative to another fixed plane surface, at a fixed distance from it, the oil in question being in contact with and between the two surfaces. In the C. G. S. system absolute viscosity is expressed by the *poise*, which is one dyne-second per square centimeter.[1] Often the viscosity of water is taken equal to 1 at 0°C. and the viscosities of other liquids are referred to this as the unit, the values obtained being specific viscosities.

The commercial instruments which have been evolved do not give results in absolute or specific viscosities, nor are they directly comparable with them, except at very high viscosities. For the Saybolt universal instrument, the following equation gives results which are approximate, but, for accurate work, the constants of the equation should be determined by the graphical methods of Higgins:[2]

$$\frac{\mu}{\gamma} = 0.00220t - \frac{1.80}{t},$$

in which μ is the viscosity expressed in poises, γ is the density of the oil at the temperature of the experiment, and t is the time of outflow of 60 c.c. of the oil.

The commercial instruments most generally employed are as follows:

(A) The Saybolt Universal Viscosimeter.—This is used widely in the United States. The results are expressed in the time necessary for 60 c.c. of the oil at a definite temperature to flow from the instrument.

(B) The Redwood.—This instrument is used in England. The results are expressed in the time necessary for 50 c.c. of the oil at a definite temperature to flow from the instrument.

(C) The Engler.—This is used in Germany and also is

[1] Herschel, Bureau of Standards *Tech. Paper* **100.**

[2] Bureau of Standards *Tech. Papers* **100** and **112**; see also Chéneveau, *J. physique*, **7** (1917), 109. On the determination of absolute viscosities of lubricants at varying temperatures and under pressures up to 10 tons per sq. in., see Stanton, *Engineering*, **108** (1919), 520.

specified for use by certain United States Government bureaus. The results are expressed in the *Engler number*, which is the quotient of the time of the outflow of 200 c.c. of the oil at a definite temperature, and the time of outflow of 200 c.c. of water at a temperature of 68°F. (20°C.).[1]

The temperature at which it is customary to determine viscosity is either 100°, 130°, or 210°F. Specifications should call for the viscosity to be taken at or near the temperature at which the bearing works, for the reason that oils change considerably in viscosity with an increase or decrease of temperature. "The higher the temperature, the lower the viscosity—at 300°F. nearly all oils have a viscosity only a little greater than water."[2] This is represented graphically in the *Appendix*, page 356. When properly conducted, the viscosity test is of the utmost importance, but as an adjunct to other tests and when interpreted in the light of service requirements.

The Saybolt and Engler instruments are described herein because of the fact that both instruments are used rather widely in this country, and not because the Engler viscosimeter has any particular points of superiority over that of Redwood.

(A) The Saybolt Universal Viscosimeter. *Apparatus.*—Saybolt universal viscosimeter[3] (see Fig. 41); thermometers reading to 215°F. for the water-bath and oil-tube; wire gauze strainer; stop-watch; ring burner or electrical heating element; receiving vessel; cleaning plunger; and 50-c.c. pipette.

[1] For a table connecting the values of the three instruments, consult the *Appendix*, page 355.

[2] Bryan, "Practical Lubrication," *J. Am. Soc. Naval Engrs.*, Nov., **1915.** See also Bryan, *J. Am. Soc. Mech. Eng.*, **37** (1915), 293.

[3] On Herschel's standardization of the Saybolt universal viscosimeter, see Bureau of Standards *Tech. Paper* **112**, 1919.

Dimensions of the Standard Saybolt Universal Viscosimeter

Dimensions	Minimum, Cm.	Normal, Cm.	Maximum, Cm.
Diameter of outlet tube	0.1750	0.1765	0.1780
Length of outlet tube	1.215	1.225	1.235
Outer diameter of outlet tube, at lower end	0.28	0.30	0.32
Height of overflow rim above bottom of outlet tube	12.55	12.60	12.65
Diameter of container	2.955	2.975	2.995
Average head (calculated)	7.32	7.47	7.61

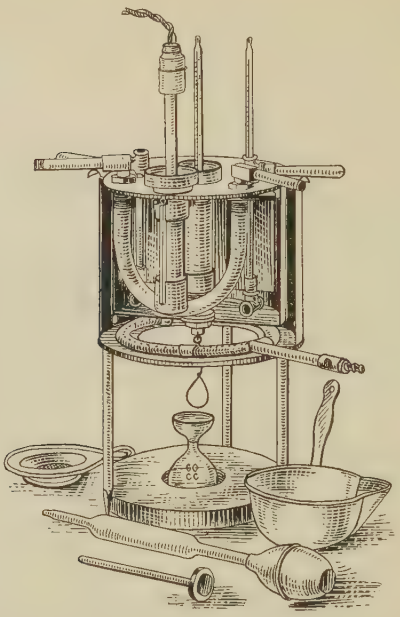

FIG. 41.—The Saybolt universal viscosimeter and accessories.

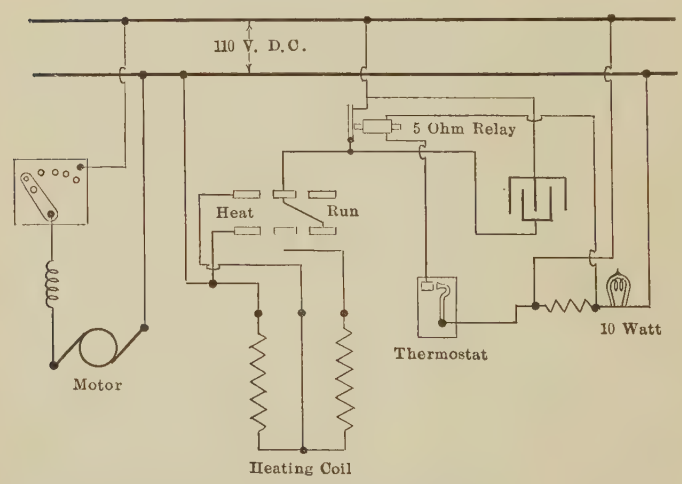

FIG. 42.—Wiring diagram for the Saybolt universal viscosimeter bath control devised by the Bureau of Standards.

Procedure.—Fill the bath with water (or pale engine oil, especially when the temperature desired is 210°F.). Pour a few c.c. of the oil into the tube through the strainer, and run the plunger up and down in the tube for the purpose of cleaning. Wipe the gallery clean with a piece of cloth. Place the cork stopper in the outlet of the oil-tube, and the receiver below the outlet. Fill the tube through the strainer with the oil to be tested so that the surface is well above the top of the tube and overflowing into the gallery. Insert the thermometers in the water-bath and the oil-tube. By means of the electrical heating element or the ring burner, heat the water (or pale engine oil) in the bath and keep it agitated by means of the handles on the cover, which are drawn back and forth. The oil in the tube is stirred occasionally with the thermometer inserted in it.[1] Remove the oil entirely from

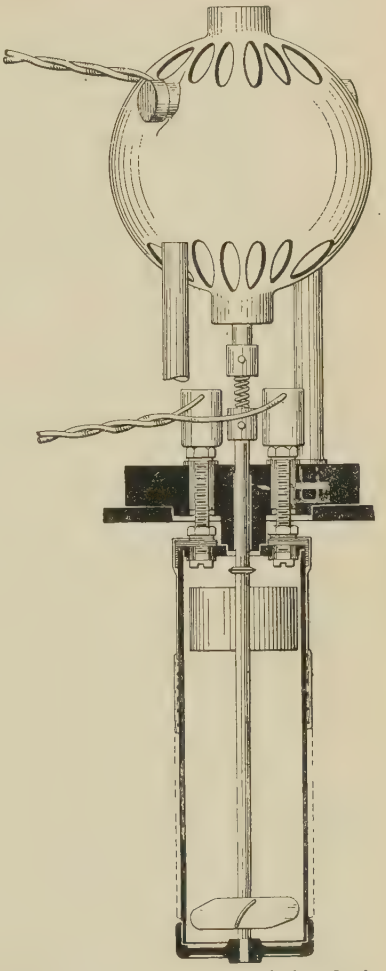

FIG. 43.—Heating and stirring device of the Saybolt universal viscosimeter bath (Bureau of Standards).

[1] The oil in the oil-tube (see *Appendix*, page 564) should not be stirred violently and the thermometer should not be allowed to rest on the bottom of the tube. When not in use for stirring the oil, the thermometer should be either suspended in the oil or removed from the tube.

The Bureau of Standards has developed a device to be attached to the Saybolt universal viscosimeter for the purpose of stirring and regulating the temperature of the bath. Fig. 42 illustrates the wiring diagram for the control of the bath, and Fig. 43 shows the heating and stirring device.

A new top plate for the viscosimeters has been used and the heating and stirring apparatus set at the left of the oil tube. The thermometer is set

the gallery on the outside of the oil-tube by means of a pipette, after *first* having regulated the temperature of the bath and of the oil in the oil-tube, to that desired. This procedure gives a constant "head" of pressure for the test.

Having the oil at the desired temperature (the determination of viscosity at 100°F. has become general in the United States, except for car oils, which are tested at 210°F. and sometimes at 130°F., and for cylinder stocks and oils, which are tested at 210°F.), remove the stopper from the outlet of the oil-tube and simultaneously start the stop-watch. Secure the time in seconds necessary to fill the receiving vessel to the mark on the neck (60 c.c.). While the oil is running out, particularly if the rate is

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directly back and the thermostat in between these two, thus leaving ¾ of the top clear for the operator.

The thermostat is a bi-metallic spiral type carried in stock by the Central Scientific Company, of Chicago, Ill. The relay is the ordinary telegraph type and the condenser is furnished with the thermostat. The motor is a K. & D. No. 44, and, because of the large change in viscosity of the bath when cold and at running temperature, it is necessary to use a rheostat in series with the motor. A six-point 500-ohm is the proper size. The heating coil is No. 30 nichrome wire of the length to give 1.2 amperes on a 110 v., wound on the brass tube with a thin sheet of mica between. The wire is wound non-inductively starting from a small fibre post at the bottom, and a No. 26 enameled copper wire is wound between the two strands of nichrome and brought up to a third post similar to those shown, for making the parallel connection for heating up on starting. The heating element is covered with a mixture of sodium silicate and alundum cement made to about the consistency of thick cream. The cement is mixed in a container the shape of the tube and after dipping the tube is put in a slow turning lathe until the mixture sets. It then is dried for several hours in an oven and a second dipping is usually necessary. This cement will not stand water, but has worked very satisfactorily in the oil-bath. The shunt which operates the relay is a piece of No. 38 nichrome, with a resistance of 15 to 20 ohms. With the low voltage this gives, the thermostat with careful adjustment will control the bath within ¼ of a degree Centigrade or better. These thermostats are not all equally good in their operation, but usually they can be adjusted to come within the limit mentioned above.

It will be evident from the drawing, what parts of the heating device are insulators. Ordinary red fiber has been used for these and redmanol would probably be equally good. This material is used for the blocks which the conductors enter and a brass cylinder is screwed down on to the ends of the wires. These cylinders fit onto split posts as shown in the drawing and the blocks have actually been made to cover the nuts shown underneath them in the drawing, so there is no chance for short circuit. On the whole, this device has worked out very successfully.

slow, the temperature of the bath should be watched and held at the requisite temperature. The results of the first run are checked by a second experiment, and the findings are expressed as Saybolt time (seconds) at the temperature of the experiment. Example: Saybolt time at 100°F. = 150 seconds.

Precautions.—Herschel[1] has found that the following points are worthy of consideration: The cork at the outlet of the oil-tube and capillary should not leak, for in that case oil will enter the space below the capillary, nor should it be inserted so far that it touches the capillary outlet. The capillary tube should be inspected often in order to see that no foreign substances have lodged therein to affect the rate of outflow; it may be cleaned by drawing fishing cord back and forth in the capillary. It is preferable to permit the stream of oil from the capillary to strike the side of the receiving vessel, so that bubbles will not be produced. The error in starting the stop watch may be large, particularly if the oil is a thin one, so that an average of several runs should be taken.

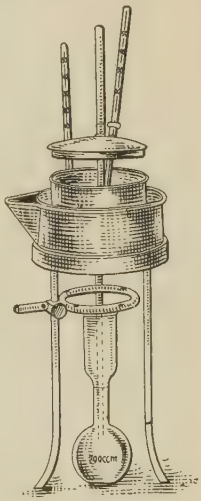

FIG. 44.—The Engler viscosimeter.

(B) THE ENGLER VISCOSIMETER. *Apparatus.*—Engler viscosimeter (see Fig. 44); thermometers for oil-cup and bath; and receiving vessel.

Procedure.—To find the water time of the instrument (which should be 50 to 52 seconds), clean the oil-cup with ethyl ether, dry carefully, swab the capillary with a small roll of paper, place the receiving vessel at the outlet of the capillary, and insert the plug. Fill the bath with water at a temperature of 20°C. and insert the thermometer in the holder. Next fill the oil-cup with water at a temperature of 20°C. until the surface of the water just touches the points of the three studs. Regulate the temperatures of the bath and of the cup so that they are both at 20°C. before making the test. The test is conducted by removing the wooden plug and simultaneously starting the stopwatch. The time (in seconds) necessary for the receiving vessel to fill to the lower mark (200 c.c.) is taken as the water time. This test is repeated until checks are had to within one second.

[1] Bureau of Standards *Tech. Paper* **100**, 1917. See also page 563.

In order to determine the *Engler number* of an oil, the cup and capillary are cleaned carefully, the receiving vessel is placed below the outlet of the capillary, the wooden plug is inserted, water (oil above 50°C.) is placed in the bath, a thermometer is inserted, and the bath is heated to the desired temperature. The oil, which has been heated to the correct temperature, is poured into the cup until the surface just touches the points of the three studs. Finally, by means of the ring burner, the temperatures of the oil and of the bath are regulated so that both are at the desired temperature before starting the test. The wooden plug then is withdrawn and, simultaneously, the stop watch is started, the time being taken for the oil surface to rise to the lower mark in the receiving vessel. This is recorded as the oil time.

The Engler number at the temperature of the experiment is calculated by dividing the time for oil by the time for water.[1]

Example: Engler number at 100°F. $= \frac{206}{50} = 4.1.$

5. Free Acid.—Sulphuric acid not removed during refining, free fatty acids present in the fatty oils used in compounding, rosin oils, or oxidation products, may be the cause of acidity in lubricating oils. In many cases, high acidity of lubricating oils will cause corrosion of journals and bearings, and first-class lubricants should be free from not only the petroleum acids and sulphuric acid, but also sulphonates. The acidity is expressed in the number of milligrams of potassium hydroxide necessary to neutralize one gram of the oil.

Method of the American Society for Testing Materials.[2] *Apparatus and Reagents.*—200-c.c. Erlenmeyer flask; burette; N/10 potassium hydroxide solution, free from carbonate; neutral 95 per cent. ethyl alcohol; and phenolphthalein solution.

Procedure.—Place 10 g. of the oil in the Erlenmeyer flask, add 50 c.c. of 95 per cent. ethyl alcohol (neutralized with weak sodium hydroxide solution), and heat to the boiling-point. Agitate the flask thoroughly, to dissolve the free fatty acids as completely as possible, and titrate while hot with the aqueous N/10 potassium hydroxide, using phenolphthalein as the indicator, agitating thoroughly after each addition of alkali. Express the result as

[1] The Engler number is not a measure of the specific viscosity.

[2] See "A. S. T. M. Standards," **1918,** 620. See also page 560.

the acid number, that is, the number of milligrams of potassium hydroxide required to saturate the free acids in one gram of the oil.

6. Sulphur and Organic Sulphur Compounds.—Little attention has been given to the *sulphur and organic sulphur compounds* which may be present in a lubricating oil. Oils intended for turbine service or in services where the lubricant is used over and over again, as in a continuous oil-circulating system, should give a satisfactorily low response when examined for sulphur content (see page 77). In carrying out the test by burning a given amount of oil in a lamp and taking up the products of combustion in an alkali solution, it is advisable, after consuming the oil in the test lamp, to make a determination of the sulphur compounds left in the wick.

Conradson[1] operates as follows:

Five to 10 g. of lubricating oil are burned to dryness in the lamp, as stated with kerosene. Spindle oils, thin turbine oils and automobile oils will feed through the wicks until they are all consumed, with a possible once or twice trimming of the crust which is formed. With thick, high-viscosity oils, 5–8 c.c. of highly refined low-sulphur kerosene are added and the mixture burned to dryness or practically so; then 2 c.c. more of the kerosene are added and burned to dryness.

The soda solution and washings are treated as described on page 78, deducting for the sulphur in the 5–10 c.c. of added kerosene.

The wicks from the lamp tests are treated as follows: Cut into small pieces, transfer to a 50 c.c. porcelain crucible, add 0.2 g. of pure dried sodium carbonate and 5 c.c. of 1.42 sp. gr. nitric acid, digest on a steam or water-bath (cover crucible with inverted lid) till the fibers are disintegrated; then add 2 g. of pure crystallized magnesium nitrate; continue the digestion, gradually raising the temperature on a hot plate or by a protected gas flame until the organic matter is destroyed and most of the nitrates decomposed, leaving a white residue; after cooling, add sufficient bromine water and hydrochloric acid, boil, dilute, and precipitate with barium chloride in the usual way and calculate to sulphuric acid (SO_3). This includes the SO_3 both in form of sulphates and sulphonates, if both are present. If it is desired to estimate the latter separately, boil the wicks with 10–15 c.c.

[1] *Cf.* WATERS, *J. Ind. Eng. Chem.*, **12** (1920), 482.

of strong barium hydroxide solution; dilute to 100 c.c. with boiling water; filter and wash. The filtrate is either oxidized with bromine and hydrochloric acid or evaporated with a few drops of nitric acid to dryness and slightly ignited, the residue being treated with hydrochloric acid and boiling water. The insoluble barium sulphate in either case is calculated to SO_3 in sulphonates. The remaining wicks with any insoluble barium salts then are oxidized with nitric acid and magnesium nitrate; the residue is treated by boiling with bromine water and hydrochloric acid; and the insoluble, if any (barium sulphate), is calculated to SO_3 present in the oil as sulphates.

7. Saponification Number.[1]—Animal and vegetable fatty oils often are added to mineral oils, especially in the case of steam cylinder oil. A lubricant for the cold parts of large marine engines is compounded of very thick blown rape oil (20 to 30 per cent.) and mineral machine oil; and additions of from 2 to 12 per cent. of neatsfoot and bone oils, as well as tallow and tallow oil, to petroleum lubricating oils are regarded as desirable for certain purposes, since the volatility is decreased and the lubricity is increased.

The percentage of fatty oil in the original lubricating oil may be found approximately by dividing the saponification number by 1.93. In order to determine the percentage of fatty oil present, a rather large quantity of the free fatty acids is separated by saponification with alcoholic potassium hydroxide, heating to expel the alcohol, extraction with ethyl ether, dilution, and finally acidification. The identity of the fatty oil then is ascertained by determining the melting-point, iodine-number, and Maumené test of the fatty acid. Having identified in this way the fatty oil present in the lubricating oil, its saponification value is found in the literature. The saponification value of the lubricating oil then is divided by this value over 100, giving the percentage of fatty oil in the original product.

Rosin oils may be recognized in mineral oils by a high acid number and by the Liebermann-Storch reaction. The last-mentioned test is carried out by agitating 1 c.c. of the oil with 1 c.c. of acetic anhydride and then separating the acid layer. A drop of sulphuric acid (sp. gr., 1.53) is added to this acid layer, whereupon a violet color will develop in the presence of rosin oil.

[1] On the determination of saponifiable oil and mineral oil and of total saponifiable matter and total fatty acids in cutting oils, see page 167.

The saponification number of a lubricating oil is determined as follows:

Apparatus and Reagents.—Burette; 200-c.c. Erlenmeyer flask; N/2 alcoholic potassium hydroxide; c.p. benzene; N/2 hydrochloric acid; and neutral ethyl alcohol.

Procedure.—3 to 8 g. of oil are weighed in the Erlenmeyer flask, 25 c.c. of the c.p. benzene and 25 c.c. of the alcoholic potassium hydroxide are added, and the whole is heated under a return condenser for 45 minutes; 50 c.c. of neutral ethyl alcohol then are added. Finally, the mixture is titrated with the standard acid, using phenolphthalein as the indicator. A blank should be run at the same time. The results are expressed in the number of milligrams of potassium hydroxide necessary to saponify 1 g. of the oil; this number is termed the *saponification number.* An improved procedure is given on page 561.

A qualitative test for the presence of a fatty oil may be made as follows: Heat a small quantity of the oil in a test-tube with metallic sodium or solid potassium hydroxide. If a fatty oil is present, in the former case the oil will gelatinize, while in the latter instance a froth will be produced on the oil, in addition to gelatinization.

8. Soaps.—Soaps dissolved in lubricating oils will cause emulsification when the oil is agitated with water, but substances other than soaps will also act as emulsifying agents. Soaps may be dissolved in an oil for the purpose of preparing a grease-base or for the preparation of emulsifiable products (on *water-soluble oils,* see page 166). Sometimes aluminum or alkali soap is added for the purpose of increasing the consistency of the oil. Such saponary oils are generally "stringy" in character and also emulsify readily, so that they are easily detected. The soaps, when present in notable amounts, may be identified by dissolving 5 c.c. of the oil in an equal part of gasoline and adding 15 drops of a saturated solution of stick phosphoric acid in absolute ethyl alcohol: if soaps are present, a flocculent precipitate will be produced.[1] The soaps may be examined further by shaking some of the oil with dilute hydrochloric acid, separating the two layers and testing the water layer for the presence of the sodium, aluminum, iron, calcium and magnesium ions.

9. Cold Tests.—Included under cold tests are *cloud* and *pour*

[1] Schweitzer and Lungwitz, *J. Soc. Chem. Ind.,* **13** (1894), 1178.

tests. These are carried out as follows, according to the methods of the American Society for Testing Materials.[1]

Cloud and Pour Tests for Petroleum Oils, Except Steam-Cylinder and Black Oils.—The *cloud test* indicates the point at which paraffin wax or other solid substances crystallize out or separate from solution in the oil. It is sometimes of value in finding the amount of paraffin present and the deportment of the oil in chilling down to a temperature above the congealing point.

Put the oil to be tested in a glass jar or bottle, approximately 1¼ in. in inside diameter and 4 to 5 in. high, to a height of about 1¼ in., or sufficient to reach ¼ in. above the mercury bulb of the thermometer. The thermometer used is the so-called cold-test thermometer, which is made especially for this purpose and has a bulb ¼ to ⅜ in. long. Insert the thermometer through a tight-fitting cork so that it is held centrally in the jar, with the lower end of the bulb ½ in. from the bottom of the jar. Then place the cold-test jar in a metal or glass jacket, 4 to 5 in. high, having an inside diameter ½ in. larger than the outside diameter of the test jar. A disk of felt, cork or wax, ¼ in. in thickness, is placed in the bottom of the jacket. Care should be taken that the test jar is placed in the center of the jacket, so that it does not touch the sides at any point. Then put the whole apparatus into the refrigerating mixture and at every drop of 2°F. in the temperature, when near the expected cloud test, remove the jar from the jacket and inspect, being careful not to disturb the oil by moving the thermometer or otherwise. When the lower half of the sample becomes opaque through chilling, read the thermometer. This reading is taken as the *cloud test* of the oil.

The *pour test* indicates the temperature at which a sample of oil in a cylindrical form of specified diameter and length will just flow under specified conditions. It is of importance in connection with the service utility of the oil and should be considered carefully.

In making this test the same bottle and quantity of oil are used as for the cloud test, and the pour test may, if desired, be made after the cloud test has been determined; in the great majority of cases the cloud test is the higher. In making the pour test, place the jar containing the oil in a close-fitting metal jacket

[1] "A. S. T. M. Standards," **1918,** 617. On the freezing-point of mineral oils, see Lantos, *Chem.-Ztg.*, **43** (1919), 853.

provided at the bottom with a disk of felt or cork, ¼ in. thick. Place this in the freezing mixture. At each drop in temperature of 5°F. remove the bottle from the jacket and tilt it until the oil begins to flow. The bottle is tilted just sufficiently, but no more; in the extreme case, it should be tilted to the horizontal. When the oil has become solid around the thermometer and will not flow, the previous 5° point shall be taken as the *pour test* of the oil.

It is preferable that the cold be applied in such a manner that the pour test will be completed in approximately one-half hour. The materials used in the freezing mixture may be ice and calcium chloride or sodium chloride, or solid carbon dioxide with acetone, depending upon the temperature desired in making the test. For oils congealing or solidifying above +35°F., pounded ice is used. For oils congealing or solidifying from +35° to +15°F., a mixture of pounded ice and a small addition of salt (sodium chloride), 1:20 by volume, may be taken. For temperatures from +15° to −5°F., an ice-and-salt mixture, containing about one-third salt, is used. The salt should be very dry and granulated fine enough to pass through a 20-mesh sieve. From zero to −25°F., a mixture of ice and calcium chloride is employed. For temperatures lower than −5°F., however, it will be found very convenient to use solid carbon dioxide and acetone, by which any desired temperature down to −70°F., or even lower, can be obtained. This freezing mixture is made as follows: Take a sufficient amount of dry acetone and put it into a covered metal beaker, copper or nickel; put the beaker into an ice-salt mixture, and when the temperature of the acetone reaches +10°F., or below, add by degrees solid carbon dioxide, until the desired temperature is reached. To get the solid carbon dioxide, take an ordinary cylinder of liquefied carbon dioxide and invert it; then open the valve carefully, and let the liquid run out into a chamois-skin bag. By the rapid evaporation which ensues a portion of the dioxide becomes solidified.

Cold Test for Steam-Cylinder and Black Oils.—The object of the cold test is to determine the lowest temperature at which an oil will flow from one end of a container to the other, in case it should become frozen and the resulting solid oil stirred until it has assumed a sufficiently pasty consistency to flow. The test is conducted by freezing solid an ounce of the oil in an ordinary 4-oz. oil-sample bottle, using a freezing mixture if neces-

sary. Then a thermometer should be introduced into the frozen mass, and, after it has become cold, the bottle containing the congealed oil is removed from the cooling medium. The frozen oil is stirred thoroughly with a thermometer until the mass will run from one end of the bottle to the other, and at this moment the temperature as indicated is recorded. The reading is the *cold test* of the oil.

If the figures indicating the cold test are inside the bottle and covered by the softened oil, the reading can be obtained by grasping the bottle by the neck with one hand, having in the same hand a piece of waste, which encloses the thermometer. The thermometer then is withdrawn rapidly through the waste and the temperature is noted.

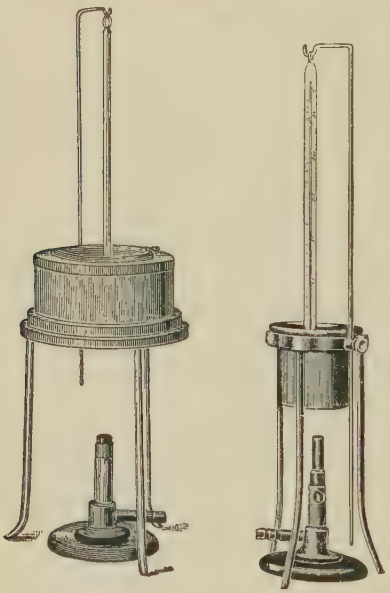

FIG. 45.—The Cleveland open-cup fire-tester, large and small sizes. The large size is the type referred to in the text (see also Fig. 34).

10. Flash and Fire Tests.—Everything else being equal, the oil with the higher flash- and burning-points should give better results in practice, as this would be an indication that the oil had been "closer cut." Too low flash and fire tests may also be objectionable because of too large a loss from evaporation in service. As a matter of safety, the flash-point should be above the working temperature of the bearing. In connection with the viscosity, cold test and gravity, the flash and fire tests enable the petroleum chemist to form an approximate idea of the source of the product under examination.

In determining the flash- and burning-points, in some laboratories a small porcelain dish ($2\frac{3}{4}$ in. by 1 in.) is imbedded in sand and filled $\frac{3}{4}$ full with the oil to be tested. The thermometer is suspended in the center of the dish with the bulb well covered by oil. The heating is done with a bunsen burner from below. The test flame is not longer than $\frac{1}{4}$ in. The rate of

heating lubricating oils should be from 10° to 14°F. per minute after arriving within 35°F. of the flash-point, but below that point it may be more rapid.

However, the Cleveland open-cup tester generally is used for determining the flash- and fire-points of lubricating oils. This apparatus (see Fig. 45) consists of a brass cup holding 100 c.c., supported in an outer vessel, with an air space between. The thermometer is suspended freely in the oil under test, so that the bulb is covered. The cup is filled to ¼ in. from the top, and the oil is heated at the rate of 10°F. per minute. As the flash-point is approached, the test flame (5 mm. in length) is moved slowly back and forth, so that the top of the flame comes within 2–3 mm. from the surface of the liquid, without, however, touching it or the sides of the container. This is repeated every 2°F. rise in temperature until the vapors flash. This tester also is used in the examination of bituminous materials.[1]

11. Carbonization. Heat Test and Carbon-Residue Test. —The heat test generally is conducted by simply heating the oil on a sand-bath in an Erlenmeyer flask at the flash-point for fifteen minutes and observing the change in color of the oil. All else being equal, the best oil is the one which changes color the least by this treatment. While there always will be some change in color, the oil should not turn black. Valuable data thus are obtained regarding the applicability of an oil for air compressors, turbines, etc.

Waters[2] carries out the test by heating the oil to 482°F. (250°C.) in an air-bath for three hours, finally determining the percentage insoluble in petroleum ether. This test is useful in the comparison of oils.[3]

[1] See page 156.

[2] *J. Ind. Eng. Chem.*, **8** (1916), 587. *Cf.* pages 106 and 561.

[3] The Waters oxidation oven has been tested thoroughly by the U. S. Bureau of Standards. The oven as used by Waters is unsuited for commercial employment because the thermostat is too fragile; accordingly, the Bureau has investigated various other types, but so far has not recommended any standard form of oven for making the test. The oven designed by Stratford ("Veedol" pamphlet, 59) has been used, but it is not entirely satisfactory. It is believed that such a type of oven is needlessly expensive and tests made with an oven constructed from a 6-in. hot-plate look quite promising. A simple structure is built upon the plate to hold six 50-c.c. Erlenmeyer flasks, the flasks being supported so as to be heated only by radiation.

Carbonization Test on Air Compressor Oils.—Weigh about 13 g. of the sample in a tared platinum dish, place in a double-lined copper oven which has been heated to 425°F. and maintain at this temperature for 6 hours. The oven should be connected with a suction-line, so that the vapors can be carried off without igniting. Cool and weigh. The loss in weight divided by the weight of the sample and multiplied by 100 gives the percentage of volatile material. Dissolve the oil in gasoline and filter through a Gooch crucible which has been prepared with a tight asbestos felt. Dry and weigh. Ignite the residue in the crucible; cool and weigh. The difference between these two weights, divided by 100, gives the relative amount of carbonization.

The *carbon residue test* is carried out by distilling 25 c.c. of the oil from a hard glass or, preferably, a fused silica flask[1] having a bulb 1¾ in. in diameter and the outlet tube about 1 in. from the top of the bulb.[2] The distillation should be conducted at the rate of a drop a second and continued until the flask goes dry. Finally, the flask is heated strongly until all oil is driven out. Any oil and carbon in the side-tube and the neck above it are burned out after the cork is removed. The flask next is cooled and weighed, and the percentage of carbon residue is calculated. This method of Gray may or may not give results which will indicate the carbonization value of the oil when used in an internal combustion engine, but the value is useful in the case of oils which have been loaded with soaps or cylinder stock for the purpose of increasing the viscosity. The carbonization value should not exceed 1 per cent.

The Conradson Apparatus and Method for Carbon Residue.—Fig. 46 shows the arrangement of the apparatus for this test. It consists of the following: (*a*) Porcelain crucible, wide form, glazed throughout, 25 to 26 c.c. capacity, and 46 mm. in diameter. (*b*) Skidmore iron crucible, 45 c.c. (1½ oz.) capacity, 65 mm. in diameter, 37 to 39 mm. high, with cover, without delivery tubes, and one opening closed. (*c*) Wrought-iron crucible with cover, about 180 c.c. capacity, 80 mm. diameter, and 58 to 60 mm. high. About 10 mm. of sand are placed in the bottom to bring the Skidmore crucible nearly flush with the top. (*d*) Triangle, large size, pipe-stem covered, with a projection on

[1] Weighed before and after the oil is poured in.

[2] The Gray carbon-residue flask and method.

its side to allow the flame to reach all sides of the crucible. (*e*) Sheet-iron hood, 5 in. in diameter and 2 in. high, provided with a slanting roof, ¾ in. high, terminating into a chimney, the chimney being 2 in. high and 2⅛ in. to 2¼ in. in diameter. This serves to distribute the heat uniformly. (*f*) Asbestos or hollow sheet-iron block, 6 to 7 in. square, and 1¼ in. to 1½ in. high, provided with an opening in the center which is 3¼ in. in diameter at the bottom and 3½ in. in diameter at the top of the block. This block acts as a shield for the flame and hence results in the even distribution of the flame around the iron crucible during the test. (*g*) Tripod or stand, which should be of such a height that the distance between the top of the burner and the bottom of the large iron crucible is 1 in. to 1½ in., depending upon the kind of burner used. (*h*) Gas burner; where gasoline or artificial gas is used, the Meker or "Scimatco" burner, 155 mm. in height and having 24 mm. section of flame, is recommended. With natural gas the above burners or any improved form of bunsen burner may be used.

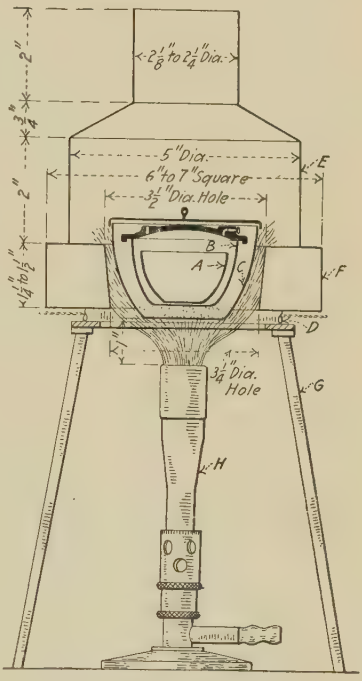

FIG. 46 —The Conradson carbon residue apparatus.

Procedure.[1]—Weigh 10 g. of the oil to be tested into a tared porcelain crucible, and place the latter in the center of the Skidmore crucible. Place the Skidmore crucible in the exact center of the iron crucible, and put on the covers of the Skidmore crucible and the iron crucible.

Heat from a bunsen or other burner is applied with a high flame surrounding the large crucible, as shown in Fig. 46. Continue heating intensely from four to seven minutes, depending upon the body of the oil under test. Then reduce the flame

[1] See CONRADSON, *Orig. Com. 8th Intern. Cong. Appl. Chem.*, **1**, 131; and *J. Ind. Eng. Chem.*, **4**, 903; see also "A. S. T. M. Standards," **1918**, 620. Cf. SOUTHER, *Proc. Am. Soc. Test. Mat.*, **8** (1908), 594.

of the burner to about 2 in. to 3 in. in height, until oil vapors begin to ignite over the crucible. Then regulate the heat so that the oil vapor (flame) comes off at a uniform rate. (The oil vapor flame should not be over 2 in. above the top of the chimney.) After the vapors cease to come off, increase the heat as at the start and maintain for five minutes, making the lower part of the large iron crucible red hot; then remove the burner, allow the apparatus to cool for five minutes before uncovering the crucible, remove the porcelain crucible, cool in a desiccator, and weigh. The entire process should be completed in about one-half hour when the heat is properly regulated. The time varies somewhat with the kind of oil tested; *e.g.*, a very thin oil with a low flash-point will not take as long as a heavy thick oil with a high flash-point.

It has been found that, if the apparatus is heated thoroughly before the porcelain crucible containing the weighed oil is placed therein, the regulation of the oil vapor (flame) may be facilitated so that it is given off uniformly and not too rapidly at first. To prevent the oil from boiling over in the porcelain crucible, because of overheating, it is very important that the first appearance of the oil vapors should be watched for very closely, and the heat regulated accordingly.

12. Volatility.[1]—An oil which will have appreciable volatilization at the temperature at which the bearing works will change considerably in viscosity during use. A useful test, but one on which too much stress should not be placed, is to heat equal quantities of two oils to be compared, for several hours in an oven at a temperature varying from 300°F. for lighter oils, to 450°F. for the heavier ones. The containing vessels should be of identical size and shape. Other conditions corresponding, it may be stated that the oil showing the smaller loss in weight by this treatment is the better.

In some laboratories the "evaporating test" is carried out by exposing the oil in a shallow flat-bottomed dish in an air-bath at a temperature of 212° to 300°F. for six hours. The percentage of loss is noted, then the condition of the residuum and its behavior when mixed with 88° Bé. gasoline.[2] If an oil gives a clear solution with this gasoline before it is heated, but affords a precipitate with it after the "evaporating test," this indicates the

[1] On evaporation tests of lubricating oils, see CONRADSON, *Proc. Am. Soc. Test. Mat.*, **18** (1918), I, 323.

[2] 5 c.c. of the oil should be mixed with 95 c.c. of the gasoline.

presence of compounds which are rather readily (depending upon the temperature of the test) acted upon by heat, and such an oil in comparison with another oil, other things being equal, would not possess the same lubricating value in service. This "gasoline test" also is applied at times to lubricating oil before and after it is heated to take the burning-point; it may be said to indicate the presence or absence of tar and asphaltic matter.

O'Neill[1] conducted an interesting investigation of the distillation of lubricating oils. He distilled the oils in superheated steam and plotted the viscosities of fractions secured against temperatures. Samples of the original oils then were placed in actual service, wherein it was found that those oils which were "close-cut" gave the best results and changed the least in the course of time. In other words, the oils having low viscosity fractions in their make-up volatilized to some extent, while those having exceedingly high viscosity fractions in their composition carbonized because of cracking of the high-boiling fraction.

13. Emulsification.[2]—For lubricating oils which are not used in forced lubrication and motor cylinders, the general preference is for oils which will emulsify with water. For forced feed, however, the use of non-emulsifiable oils is essential, since a permanent emulsion can not be separated readily in the filtering and settling chamber. An oil which will emulsify with water contains some substance which acts as the emulsifying agent. Examples of emulsifiers are salts of organic acids, such as the calcium and magnesium salts of palmitic and stearic acids, sulphonates and the salts of naphthenic acids.

The resistance of an oil to emulsification is known as the *demulsibility.* Two procedures have been proposed by the determination of the demulsification value, *viz.*, the Bureau of Standards method and the Conradson method. In the first-mentioned

[1] *J. Am. Soc. Naval Eng.*, May, **1916**; *Power*, **44**, 108; *Sci. Abstracts*, **19B**, 317.

[2] For an interesting discussion of the modern conception of emulsions, see CLAYTON, *J. Soc. Chem. Ind.*, **38** (1919), 113T. BANCROFT's papers on the theory of emulsification are also valuable (see *J. Phys. Chem.*, **16**, 177, 345, 739; **17**, 501; **19**, 275, 513; and **20**, 1). See also LEWIS' "A System of Physical Chemistry," 2nd ed., **1**, 19, 326, 353 and 368; and THOMAS, *J. Ind. Eng. Chem.*, **12** (1920), 177.

method,[1] the oil (20 c.c.) and the water (40 c.c.) are stirred in a 100-c.c. cylinder at 1500 r.p.m. for five minutes at a temperature

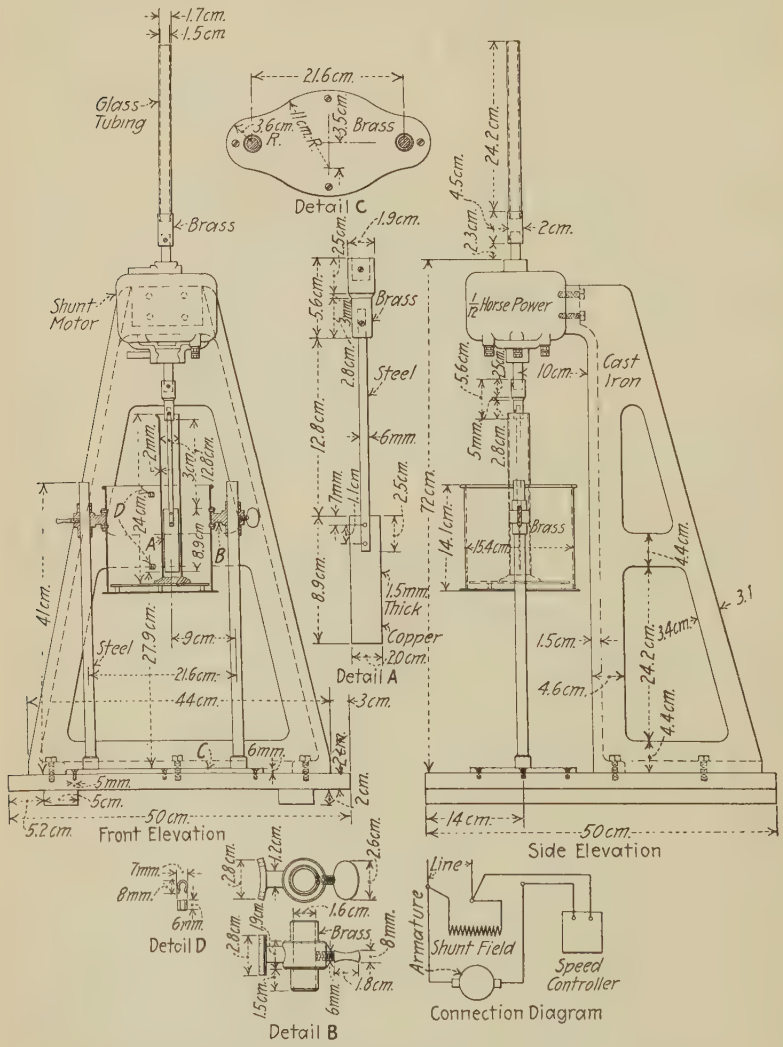

FIG. 47.—Emulsifier for applying the demulsibility test on lubricating oils (Bureau of Standards).

of 55°C. (131°F.). The stirrer is a rod having at one end a metal

[1] Bureau of Standards *Tech. Paper* **86**; *Proc. Am. Soc. Testing Mat.*, **19** (1916), II, 259. See also STRATFORD, *Motor*, Nov., **1916**, 92; and *Motorboating*, Dec., **1916**, 34.

plate, 89 by 20 by 1.5 mm. The cylinder is allowed to stand for a period of time not exceeding one hour in length, at a temperature of 55°C. (131°F.); and from each of the readings, taken as frequently as necessary, of the volume of oil settled out from the emulsion, there is calculated the average rate of settling between stopping the paddle and the time of observation. The maximum rate of settling thus obtained is termed the demulsibility, and is used as a measure of the resistance of the oil to emulsification. The maximum possible demulsibility is 1200, as the first reading is taken one minute after stopping the paddle. Example: After 10 minutes, the reading of the meniscus was 40 c.c.; therefore,

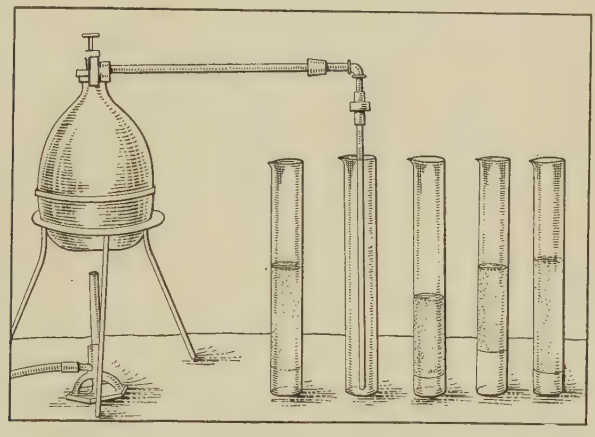

FIG. 48.—Conradson oil-emulsification testing apparatus.

the volume of oil separated was 60 − 40 c.c., or 20 c.c., and the rate of demulsibility is 20 : 10 : : X : 60, which equals 120 c.c. per hour.

The method of Conradson is as follows:[1]

Apparatus.—Fig. 48 requires but little explanation. The apparatus consists of a 4-pint copper retort, provided with a delivery tube, which is joined to a metal or glass pipe having an inside diameter of about $\frac{5}{16}$ in. and about 15 in. long from the elbow. The lower end of this pipe is cut off diagonally to prevent thumping. The glass cylinders are graduated to 250 c.c. They have an inside diameter of about $1\frac{7}{16}$ in. and a length of about $9\frac{1}{2}$ in. from the bottom to the 250-c.c. mark. They are $11\frac{1}{2}$ to

[1] *Proc. Am. Soc. Test. Mat.*, **16** (1916), II, 273.

12 in. in overall length, and are made of thin glass, with a flat bottom.[1]

In place of a copper retort for the generation of steam, a glass flask or any other suitable source of steam supply may be used; likewise, ordinary 250-c.c. graduated glass cylinders having the dimensions given above, may be used where emulsion tests are required only occasionally.

Method of Testing.—The cylinder is filled with distilled water up to the 20-c.c. mark, then 100 c.c. of the oil to be tested are added. To churn the mixture, steam at ordinary pressure is conducted through this oil-water mixture for ten minutes. The amount of steam passed through is regulated in such a way as to prevent the mixture from splashing over the top of the cylinder, but the rate may be as rapid as is practicable. This is regulated by the height of the gas flame. The churning is begun from the time the temperature of the mixture has reached 200°F., or when the steam as such passes off the mixture. It usually takes from 1 to 1½ minutes to reach this temperature, depending somewhat on the body or viscosity of the oil. However, even churning with steam for 15 minutes does not seem to make any difference in the results.

When the churning is completed, the cylinder is immersed for one hour in a water-bath, kept at a temperature of 130°F. During this time the cylinder and its contents are inspected momentarily at intervals, to note the behavior of the oil mixture. At the expiration of one hour the cylinder is removed from the water-bath and the contents are examined for the following facts:

(*a*) The number of cubic centimeters of separated clear or turbid water;

(*b*) The number of cubic centimeters of separated emulsified layer;

(*c*) The number of cubic centimeters of separated clear or turbid oil above the emulsified layer; and

(*d*) The percentage of water or moisture in the separated oil above the emulsified layer.

The number of cubic centimeters and condition of the emulsified layer constitute an indication of the emulsion-forming property or quality of the oil.

The number of cubic centimeters of clear or turbid oil above

[1] The glass cylinders used for carrying out duplicate tests should be of exactly the same dimensions.

the emulsified layer, less the percentage of water or moisture contained in the oil, is the percentage of demulsibility of the oil. The condition of the separated water or watery liquid under the emulsified layer, if any, gives an indication also of the behavior of the oil in actual service.

The amount of water held in the oil above the emulsified layer may be determined as follows: The oil above the emulsified layer, after the completion of the test, is drawn off carefully and shaken; then 20 c.c. are mixed with 80 c.c. of 88° Bé. gasoline (from Pennsylvanian crude) in a graduated, flat-glass precipitating tube having the lower end drawn out. The oil-gasoline mixture is kept at a temperature not over 80°F. for one hour, or the water or watery liquid may be separated from the oil-gasoline mixture by means of a centrifuge. The amount of water or watery liquid is read off and calculated to percentage by volume and subtracted from the oil above the emulsified layer. Of course, this determination is only necessary when the oil above the emulsified layer appears to contain an appreciable amount of water.

Interpretation of Results.—Fig. 49 illustrates the behavior of seven representative oils with this method, as they appear after the conclusion of the tests. The following table gives the detailed results of tests on these oils. The first turbine oil[1] shows

EMULSIFICATION TESTS OF LUBRICATING OILS

Oil No. Kind of oil	Separated water in c.c.	Condition of water	Emulsified layer in c.c.	Kind of emulsion	Separated oil in c.c.	Condition of oil	Water in oil in per cent.	Demulsibility in per cent.
1. Turbine.....	40	Clear	None	.....	100	Slightly turbid	0.2	99.8
2. Turbine.....	36	Slightly turbid	12	Light, foamy	100	Turbid	1.0	99.0
3. Crank-case..	40	Clear	None	.....	105	Very turbid	5.0	95.0
4. Crank-case..	4	Clear	150	Heavy, thick	Trace	Practically all emulsion	.....	0.0
5. Engine.....	None	.....	50	Thick, milky	102	Very turbid	4.0	96.0
6. Engine.....	22	Clear	28	Light, foamy	102	Turbid	3.0	97.0
7. Spindle.....	20	Milky	140	Thick, milky	None	All emulsion	.....	0.0

[1] The emulsifying test is used for determining the adaptability of an oil for lubrication in turbines of the Curtis type; it is essential to learn the behavior of the oil when coming in contact with steam. On turbine oil testing, see also FRANK, *Z. angew. Chem.*, **32** (1919), 374.

the oil entirely free from emulsifying property or substance, the sample retaining but a very small percentage of water after the expiration of the test. The second turbine oil shows quite a little emulsified layer, but the condition of the emulsified layer is light and foamy, not compact or creamy. The amount of water retained in this oil is much higher than in the first oil.

Consider next the two samples of crank-case oil: The first oil shows ready separation of water, which is clear and has no emulsified layer, but the oil after the test retains about 5 per cent. of

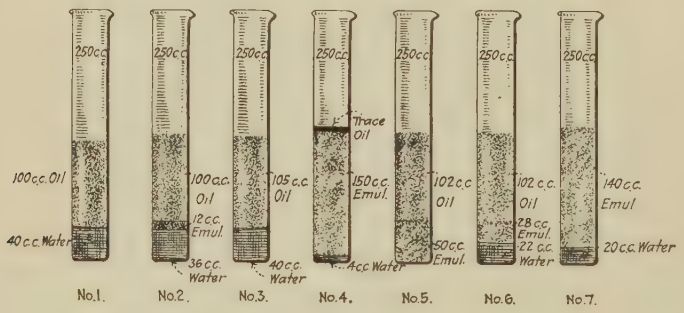

FIG. 49.—Representative lubricating oils at the expiration of emulsification tests.

water. With the other crank-case oil only a very few cubic centimeters of water are separated at the expiration of the test, and a very large amount of emulsified layer of a heavy thick nature is shown; in fact, the whole mixture is a heavy emulsion without separation.

The first sample of engine oil shows at the end of the test a thick milky emulsion with practically no separation of water, and the separated oil above the emulsified layer contains about 4 per cent. of water. The second sample of engine oil shows a considerable amount of separation of water and a much smaller amount of emulsified layer; this layer is of a light foamy nature. It should be noted particularly in these two cases that while 100 c.c. of oil were used in the tests, 102 c.c. of separated turbid oil were found; deducting the amount of water found in the separated oil, 4 and 3 per cent., respectively, gives 96 and 97 per cent. of demulsibility. This is a clear illustration of the importance of giving a complete statement in reporting the behavior of an oil or of oils in the emulsifying test; simply indicating the per-

centage of demulsibility is clearly insufficient, and in cases of this kind would be seriously misleading.

14. Maumené Number.—The specific temperature reaction or Maumené number[1] will be higher for oil compounded with vegetable or animal oils than for the pure mineral oil. Mineral oil which has been heated in air for a time also will have a higher Maumené number than the original.

The test is carried out very simply as follows: 50 g. of oil are placed in a jacketed 150 c.c. beaker; 10 c.c. of concentrated sulphuric acid are added from a pipette, drop by drop with stirring, and the temperature is noted during the process. The highest temperature reached, minus the temperature of the oil at the start of the test—*i.e.*, the rise of temperature, expressed in degrees C.—gives the Maumené number.

15. Special Tests on Valve and Cylinder Oils.—The introduction of a high degree of superheated steam in locomotive and stationary engine practice has brought about the necessity of adding tests to the ones usually made on cylinder oils, whether straight petroleum stock, or mixtures thereof with smaller or larger amounts of saponifiable fats.

Conradson has constructed and used an apparatus for some time in the investigation of cylinder oils suitable for the lubrication of valves and cylinders in connection with the use of highly superheated steam in locomotive service.[2] The apparatus is essentially as follows (see Fig. 50):

A, small steam-boiler with gas, steam gages and pipe connections. *B*, superheater with armored thermometer, *T*, and steam connections (a small circulating water-heater with double copper coils, answers very well). *C*, sight-feed lubricator with connections. *D*, steam vessel (a conical cast-iron retort for distilling mercury, about 95 mm. deep and about 75 mm. in diameter, at top, provided with two strong screw clamps to keep the lid tight, answers), surrounded on the outside, as indicated, by a coil of copper pipe, 5–6 mm. inside diameter, for further heating of the steam and gases used in the tests; this coil is tapped into the bottom of the steam vessel containing a suitably

[1] MAUMENÉ, *Compt. rend.*, **35** (1852), 572; THOMSON and BALLANTYNE, *J. Soc. Chem. Ind.*, **10** (1891), 234; and SHERMAN, DANZIGER and KOHNSTAMM, *J. Am. Chem. Soc.*, **24** (1902), 266.

[2] *J. Ind. Eng. Chem.*, **4** (1912), 744; and *Proc. Western Railway Club*, **24** (1912), 164.

supported dish of about 25 c.c. capacity (an ointment box about 55 mm. in diameter and 18–20 mm. high), into which the oil to be examined is fed drop by drop from the sight-feed lubricator *C* (or a given amount of oil is poured into the cup I). Through the lid is inserted an armored thermometer, *T*, reaching down into the dish. The steam vessel thus arranged and connected is surrounded and covered by a suitable asbestos hood. *L*, pipe connections for the gases used. *E*, heavy glass cylinder, about 200 mm. long and 18–20 mm. inside diameter, containing a small rectangular boat or dish; beyond *E* is a glass tubing system, *F*, connected up as shown with brass fittings; each glass tube is about 500 mm. long and 8–9 mm. inside diameter; at the further

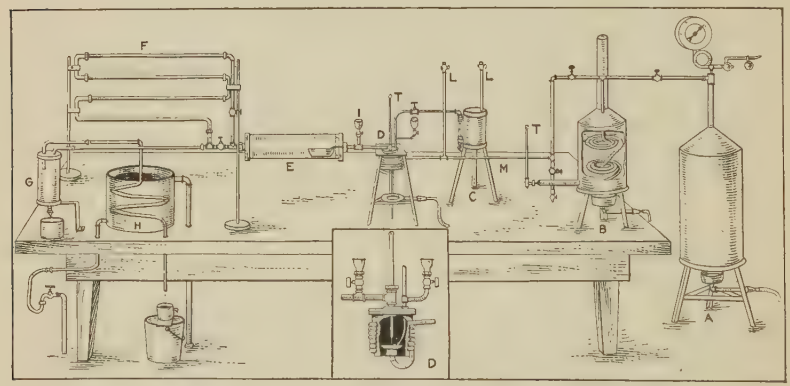

Fig. 50.—Conradson's apparatus for use in the investigation of cylinder oils.

end is attached a steam separator and oil-collecting vessel, *G*, consisting of an ordinary glass chimney fitted up with brass caps; to this the condenser *H* is connected by means of copper tubing.

In this apparatus any desired temperature may be maintained, at least up to 1000°F.; this is 300°–400° higher than is required under the most exacting service conditions, as far as superheated-steam temperature is concerned.

In studying the behavior of valve and cylinder oils in an atmosphere of superheated steam, the steam is passed from the small steam boiler through the superheater *B* and into the steam vessel *D* through its pipe coil (lamp being lit under *D*), and out through *E*, *F*, *G* and *H*. When the temperature reaches and is regulated in *D* to 400°F., oil is fed drop by drop from the sight-feed lubri-

cator *C* (or the cup *I* is filled); the steam is shut off and the valve in *C* (or *I*) opened, letting the oil run into the dish in *D*; the steam valve then is opened very gradually and carefully so as to let through any desired amount of steam. The temperature in *D* is maintained for a given length of time at or near 400°F., noting if any oil vapors are coming over; by shutting and opening the steam valve, it is easy to see whether any vapors are coming over with the steam. The temperature is increased in increments of 25°–50°F., at a time, until the desired temperature is reached, noting the behavior of the oil vapors passing on with the steam through the glass pipe system, etc. It is interesting to note that cylinder oils containing rather a large percentage of saponifiable fats or fat oils generally come over at much lower temperatures than the main portions of the petroleum stock oils that are used commonly in compounding first-class cylinder oils.

In further study of the behavior of oils, the boat in the glass cylinder *E* may be filled through the cup *I* to the left, and subjected at various temperatures to slow currents of superheated steam, carbon dioxide and air, or mixtures thereof. The glass cylinder *E* during the last test is surrounded by a heavy asbestos covering to prevent radiation.

The cylinder oils may leave a residue in the dish in *D* at steam temperatures below 700°F.; if so, such a residue should give a clear solution in 90 c.c. of 0.65 sp. gr. (87°Bé.) petroleum ether (Pennsylvanian) and show no precipitate on standing. At steam temperatures of 850°–900°F., all the oil usually has volatilized with the steam; good oils should leave no carbonaceous or coky residue.

While this apparatus was designed principally for the study of the behavior of valve and cylinder oils in atmospheres of steam at various temperatures, it can be adapted very readily for the investigation of other oils at various temperatures in currents of air, carbon dioxide or other gases, or mixtures thereof with and without steam.

The accompanying table of comparative tests of five samples of cylinder oils, *A*, *B*, *C*, *D* and *E*, and *F*, a petroleum distillate of interest in connection with the study of cylinder oils in superheated steam, has been prepared by Conradson. Hot-air test = 13 g. of oil in shallow, round flat-bottom iron dishes, exposed six hours at 540°F. in a specially designed air-bath. Gasoline test = 10 c.c. oil, 90 c.c. petroleum ether [0.65 sp. gr. (from Pennsyl-

vanian crude)] in graduated flat precipitating tubes, taking the reading after standing for one hour. Carbon test using 35 g. of oil, according to Conradson's apparatus and method. Superheated steam test = 13 g. of oil used.

	A	*B*	*C*	*D*	*E*	*F*
Flash-point (open-cup), deg. F.	545	550	605	595	550	365
Burning-point, deg. F.	610	630	695	680	630	415
Baumé gravity at 60°F.	26.4	26.1	24.7	25.9	...	33.5
Sp. gr. at 15°C.	0.895	0.897	0.905	0.898	...	0.856
Color	Light	Dark	Dark	Very dark	...	Yellow
Gas, test before fire-test	Good	Good	0.5 c.c.	4.0 c.c.		
Gas, test after fire-test.	Good	Good	Good	3.0 c.c.		
Cold-test flows, deg. F.	+55	+32	+45	+34	+30	Zero
Saponifiable fats	Trace	Trace	Trace	Trace	15	None
Vis. Saybolt at 212°F. (60 c.c.), seconds	133	146	215	216		
Barbey Ixomètre, 500°F. (180 units = 30 c.c.), seconds	45	51	61	60		
Hot-air test at 540°F. (loss), per cent	15	11	2.5	5.5		
Gas, test after	Good	Good	0.5 c.c.	3.5 c.c.		
Carbon-test residue, per cent	2.15	2.70	4.90	5.10		
SO_3 in residue, per cent.	0.023	0.03	0.03	0.04		
Loss in superheated steam at:						
400°F., per cent.	0.0	0.0	0.0	0.0	0.0	32
500°F., per cent.	5.0	4.0	1.5	1.0	5.0	67.0
600°F., per cent.	18.5	18.0	6.5	8.5	21.5	
700°F., per cent.	44.0	34.0	32.5	40.5		
Total loss up to 700°F., per cent.	67.5	56.5	40.5	50.0	26.5	99.0
Gas test of oil:						
Residue from 700° test	Good	Good	1.5 c.c.	3.0 c.c.		

Sample "*A*" in a superheated-steam test at 800°F. (427°C.) left no residue. Sample "*C*" left 2.5 per cent. of dry carbonaceous residue.

Sample "*E*" containing 15 per cent. of saponifiable fats, sub-

jected to the superheated-steam test, lost 26.5 per cent. up to 600°F. The oil residue from this test contained 17.5 per cent. of saponifiable fats; this indicates that the petroleum oil stock ("*B*" used) goes off with the steam somewhat faster in proportion to the fat oil up to 600°F. (350°C.)

The steam pressures used in these tests were about ten to twelve pounds per sq. in. A large volume of superheated steam passed through the apparatus during the tests (about 40 c.c. condensed steam per minute).

In these superheated-steam evaporating tests about 13 g. of oil were weighed into the small dish placed inside the steam vessel *D*. (The capacity of the small iron dish is 50 c.c.; it has a diameter of about 48 mm. and is 30 mm. high, with flat bottom.)

The steam vessel with the oil in the dish was heated up to about 350°F. (176°C.), passing a slow current of natural gas through the apparatus, then superheated steam was admitted, the gas shut off, and the temperature raised up to the required degree and kept constant for about 75 minutes; the volatile matters in the oils at the given test temperature generally were carried over with the steam inside of 60 minutes, allowing about 15 minutes' extra steaming. At the end of each given temperature test, the steam and heat were shut off; after cooling, the dish containing the oil was weighed and replaced in the steam vessel, and the operation repeated for the next temperature test.

16. Lubricating Greases.—The theory of emulsification, as presented by Bancroft, is that a permanent emulsion depends upon the presence of a third substance, an emulsifying agent, which, by adsorption, collects at the surface between oil and water, there forming a film. The permanency of the film will determine the permanence of the emulsion. Greases, or "consistent lubricants," are generally colloidal solutions of soaps in petroleum lubricating oils, along with a small quantity of water dispersed through the mass.[1] The water, which is in a state of emulsification in the oil, makes possible the peculiar consistency of the product as well as prevents separation of the colloidal soap. Some greases, however, do not contain water.

Greases find industrial application with heavily loaded bearings, particularly when exposed to a temperature as high as that

[1] On the value of grease lubricants, see *Power*, Nov. 9, **1909**; see also Holde, *Z. angew. Chem.*, **21** (1908), 41 and 2138; *Kolloid-Z.*, **3** (1908), 270; and Künkler, "Die Fabrikation der Schmiermittel," 1897.

of the rolls in steel rolling mills.[1] Journal greases are usually solutions of lime (calcium) soaps (15 to 23 per cent.) in heavy mineral oil, with 1 to 4 per cent. of water emulsified in them. Straight soda greases are oils thickened by the addition of soda soap to the extent of ofttimes 50 per cent., but no water; they are better lubricants than those compounded with lime soaps. Axle greases are made by treating rosin oils with lime, afterward adding mineral oil or water, or both. Some American automobile greases consist of aluminum soaps dissolved in mineral oils with water emulsified in them by the aid of rosin. In fact, various mixtures of wool fat, tallow, alkali soaps, graphite, etc., with mineral oils are marketed. Residues from the distillation of petroleum also are used as greases, but do not stand up under heat as satisfactorily as the others do. A general method for the examination of greases is difficult to find, owing to the great differences in composition.[2]

The common test for greases is that of *melting-point*, or "dropping-point," the most important single determination. One method is to dip the bulb of a thermometer in the melted grease, and, after allowing it to stand for 30 minutes, to place the thermometer on the inside of a large test-tube, with the bulb located in the center of the cross-section and about one inch from the bottom of the tube. The apparatus then is heated from below over a piece of asbestos. The temperature at which the first drop collects on the bulb is taken as the melting-point. Another method, described by Gillett,[3] is as follows:

Apparatus.—Glass tube, 8 cm. long by 0.4 cm. in internal diameter, open at both ends; 200-c.c. beaker; thermometer; and bunsen burner.

Procedure.—One end of the tube is dipped in the grease, so that

[1] On the manufacture of saponified lubricants (lubricating greases), see Kraus, *Seife*, **3** (1919), 602, 640 and 666.

[2] For detailed methods of procedure, including the determination of consistency, melting-point, flash-point, free acid, soap, oils, filler and water, consult Holde-Mueller's "Examination of Hydrocarbon Oils," Archbutt and Deeley's "Lubrication and Lubricants," and Lockhart's "American Lubricants." Greases must be well mixed and homogeneous and should not melt under 70°C. On standing or in service they should not separate into layers or oxidize. On the consistency of greases, see Clulow and Taylor, *J. Soc. Chem. Ind.*, **39** (1920), 291 T.

[3] *J. Ind. Eng. Chem.*, **1** (1909), 351. Gillett also covers the analysis of greases. On the analysis of lime greases, see Trotter, *Mech. Eng.*, **41** (1919), 815.

a column 1 cm. long is had. Then the tube is attached to a thermometer by means of a rubber band, so that the bottom of the grease column is opposite to the thermometer bulb, and finally is placed in a beaker of water, with the bottom of the grease column just 5 cm. below the surface. The water is heated at the rate of 3–4°C. (7.2°F.) per minute. The temperature is read when the grease column moves upward in the tube. This represents the melting-point.

The melting-point of greases frequently lies between the temperatures 75° and 83°C.; in the case of rolling-mill greases it may, however, be as high as 130°C. It is dependent upon the amount of soap and of oil contained in the grease, the amount of water present, the temperature and length of heating on dissolving the soap in the oil, the viscosity of the oil used, etc.

17. The Theory of Lubrication and the Coefficient of Friction. Those who have made a special study of lubrication do not always agree as to just what defines the lubricating value of an oil. The work of Ubbelohde[1] tends to establish the fact that all oils of the same viscosity have the same lubricating power. Ubbelohde showed that mineral oils having the same specific[2] viscosities, whether refined or unrefined oils, distilled or undistilled oils, possessed identical coefficients of friction, without regard to the origin of the oils.

That theoretical principle, however, must be modified to meet practical conditions.[3] Oils of the same viscosity do not always hold their viscosity in service as might be expected, oxidation taking place and carbon and asphaltic bodies being developed in the oil. O'Neill (see page 107) has demonstrated that one oil may change less in service than another because of the fact that it is "closer cut." Lubricating oils must have peculiar properties aside from the viscosity, when used for special purposes, as in forced-feed lubrication, for example, where oils which do not emulsify with water are essential; and in the case of steam-cylinder oils, which, to da e, have been manufactured by blending heavy mineral oil residues with fatty oils, in order to

[1] *Petroleum*, **7,** 773, 882 and 938; **8,** 683; *Seifenseider Ztg.*, **39,** 1009 and 1045; and *Petroleum Rev.*, **27,** 293 and 325. *Cf.* DEELEY, *Engineering*, **108** (1919), 788.

[2] Absolute viscosity and specific viscosity are closely related (see page 90).

[3] On practical requirements, see DENTON, *Trans. Am. Soc. Mech. Eng.*, **11,** 1013; and BAILEY, *Proc. Inst. Civ. Eng.*, **45,** 372.

promote adhesion to the metal surfaces in the steam cylinders. Sometimes a lubricating oil is said to possess "body," a rather vague property, difficult to define.[1]

An interesting report on the relation between the viscosity and the chemical constitution of lubricating oils has been published by Dunstan and Thole.[2] All the experiments were carried out on six oils, both before and after refining with two volumes of concentrated sulphuric acid. In three instances the refining losses were 33.3 per cent., 20.0 per cent., and 50 per cent., respectively; these figures appear to indicate a somewhat drastic chemical treatment. In every case the refined oils possessed lower viscosities and specific gravities but higher molecular weights, than the corresponding unrefined oils, but the coefficients of alteration of viscosity with temperature remained practically unchanged. As a rule, the ratio of the percentage of carbon to hydrogen was also lower in the refined oils. One Russian lubricating oil was, however, an exception to this rule.

Although the paper of Dunstan and Thole nominally is concerned mainly with the viscosities of lubricating oils, it actually deals very largely with the consideration of their "lubricating values." Dunstan and Thole are inclined to consider that there is at present no method of determining the lubricating value of an oil; but, in spite of this, throughout their report they frequently make statements showing that one oil is a better or worse lubricant than another. It is clear that such statements must be based on the results of some measurements, and presumably these measurements either are the somewhat vague results of practical experience with machinery under running

[1] In his consideration of lubricants for internal-combustion engines, Conradson (*Proc. Am. Soc. Test. Mat.*, **18** (1918), II, 388) points out that the selection of a proper grade of oil for each class of service depends upon a number of factors, such as type and condition of machine, lubricating system, method of applying lubricant, method of cooling, and climatic conditions. On the relations of viscosity and "body," see Kingsbury, *Trans. Am. Soc. Mech. Eng.*, **1903**, 147.

By adding 1 per cent. of free fatty acid to a mineral oil the lubricating power is increased as much as by adding a very much larger percentage of fatty oil. The emulsifying properties also can be modified materially by the addition of fatty acids (*Nature*, **104** (1920), 451). On lubrication as a problem of colloid physics, see Alexander, *J. Ind. Eng. Chem.*, **12** (1920), 435. On the surface tension of oils, see Grunmach and Bein, Z. *Instrumentenk.*, **39** (1919), 195.

[2] *J. Inst. Pet. Tech.*, **4** (1918), 191.

conditions,[1] or have been obtained from tests made on some type of mechanical oil-tester.

The experience of experts on this subject has been that, when the results of the lubricating values of a large number of oils of the same class, as measured on a Thurston oil-tester at the same temperature, bearing pressure, and speed of rubbing surfaces, by an observer ignorant of the viscosities of the oils, are tabulated in their order of magnitude, they are found to have been arranged in the reverse order of the viscosities of the same oils at the testing temperature. This experience is in accord with statements of Dunstan and Thole that for lubricants the lowest viscosity compatible with circumstances is essential, because otherwise energy is absorbed needlessly and converted into heat. The lubricant is concerned with the transformation of solid into liquid friction, and therefore its own internal friction should be as near zero as possible. It is thought that some measure of the lubricating value of an oil is had in the results of tests in a mechanical tester such as that of Thurston. As to how far these results are directly proportional to the true lubricating value is perhaps not fully demonstrated, but the results obtained are thought to be of real technical value.[2] From a practical standpoint, whether the mechanically most efficient lubricant is the most economical depends somewhat upon the ratio of efficiency, the amount employed and the cost of the material.[3] Most users of lubricants eliminate the use of mechanical testers, and either purchase a definite brand of lubricating oil which has been found to be satisfactory or buy on specifications secured by service tests.

Scientific literature contains descriptions of many types of mechanical testers,[4] among which are the light and heavy types

[1] On the lubrication of machinery of all types, see BATTLE's "Lubricating Engineer's Handbook," **1916,** 126–90. For considerations of the lubrication problems found in power plants, see DEMARY, *Power Plant Eng.*, **23** (1919), 955; and JUNE, *Power House*, **12** (1919), 411.

[2] On lubrication value, see UBBELOHDF, *Petroleum*, **7** (1912), 773, 882 and 938; and PETROFF, "Theorie der Reibung," **1887.**

[3] Greases have a low mechanical efficiency compared with oils, but from the point of economy and cleanliness they are more suitable for certain uses.

[4] See, especially, for a description of various mechanical testers, ARCHBUTT and DEELEY's "Lubrication and Lubricants," 3rd ed. On machines for testing lubricants, see also THURSTON's "Materials of Engineering," 5th ed., **1,** 251; and CARPENTER and DIEDERICHS' "Experimental Engineering," 7th ed., 256. On an apparatus which measures relatively the durability and the internal friction of an oil, see MOORE and RICHTER, *Met. Chem. Eng.*, **16** (1916), 692.

of Thurston machine, the Kingsbury, Hess, Wilkins, Dettmar, Olsen-Cornell, Ashcroft, Boult, Martens, Richlé, and many others. In the Thurston machine (see Fig. 51), two brasses work against a cylindrical journal. The upper brass supports a

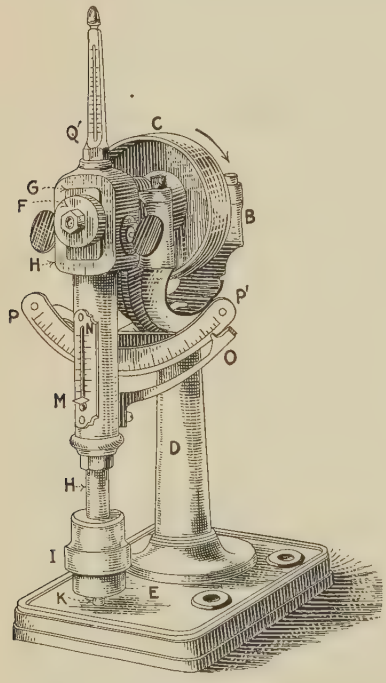

FIG. 51.—Perspective view of Thurston's oil-testing machine.

At *F* is the journal on which the lubricating material is to be placed for test. This journal is on the overhung extremity of shaft *A*, which is carried in bearings, *BB'*, on a standard, *DD'*, mounted on a base-plate, *EE'*. The shaft is driven by a pulley, *C*, at any desired speed. The testing journal, *F*, is grasped by bearings of bronze, *GG'*, and with a pressure which is adjusted by the compression of a helical spring. The total pressure on the journal and the pressure per square inch are both shown on the index-plate, *NN'*, by a pointer, *M*. A pointer, *O*, traverses an arc, *PP'*, and indicates the angle assumed by the pendulum, *H*, and ball, *I*, at any moment. The graduations on the fixed scale, *P'P*, show the total friction, *F'*; this, divided by the total pressure, *P*, gives μ, the coefficient of friction.

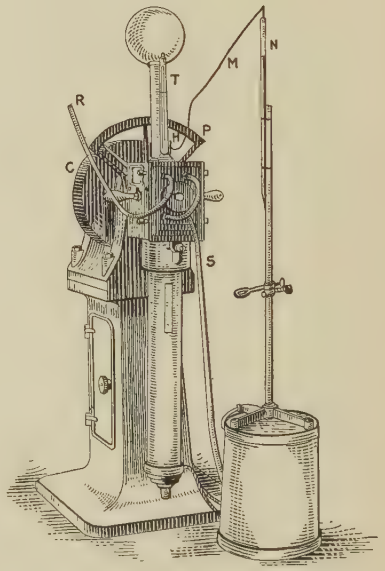

FIG. 52.—Thurston's railroad lubricant testing-machine.

The Thurston machine is made in two sizes; the larger one having axles and bearings of the same dimensions as those used in standard car construction, is shown in one form in the above cut. Explanation of symbols: *T*, thermometer, giving the temperature of bearings; *R*, *S*, rubber tubes for circulating water through the bearings; *N*, burette, furnishing the supply of oil; *M*, siphon, controlling the supply of oil; *H*, rod for receiving the oil from candle-wicking feed.

pendulum which contains a spring pressing against the lower brass. The pressure of the brasses on the journal may be increased by tightening the spring. The lubricating oil is supplied

between the revolving journal and the brasses. The coefficient of friction, μ, equals $\frac{F}{P}$.

F = total frictional resistance = $\frac{Wr \sin e}{r_o}$, where W equals the weight of the pendulum, r equals the radius from center of journal to center of gravity of the pendulum, r_o equals the radius of the journal, and e equals the angle between the pendulum and perpendicular through the axis.

P = total pressure of the brasses on the journal.

In practice F and P are secured by direct readings.

18. Specifications for Lubricating Oils.—The *Appendix*, pages 380 and 558, gives general requirements for lubricating oils.

19. Blending of Lubricating Oils.—The viscosity of a mixture of two oils is not an additive property, so that the ultimate viscosity is best determined by means of an equation[1] or, what is more practical, by the aid of a curve (see the *Appendix*, page 357).

[1] *J. Ind. Eng. Chem.*, **1** (1909), 13.

CHAPTER V

THE EXAMINATION OF BITUMINOUS ROAD MATERIALS

1. Classification of Commonly Used Bituminous Materials.
2. Scheme of Examination.
3. Specific Gravity.
4. Float Test.
5. Penetration Test.
6. Melting-Point.
7. Volatilization Test.
8. Bitumen Soluble in Carbon Disulphide (Total Bitumen).
9. Determination of Bitumen Insoluble in Paraffin Naphtha.
10. Determination of Fixed Carbon.
11. Paraffin Scale.
12. Extraction of Bituminous Aggregates; Recovery of Bitumen.
13. Grading the Mineral Aggregate.
14. Determination of Voids in the Mineral Aggregate.
15. Flash- and Burning-Points.
16. Petrolenes, Asphaltenes and Carbenes.
17. Other Tests on Bituminous Road Materials.
18. Specifications for Bituminous Road Materials, etc.

The methods included in this Chapter are predominantly those of the Office of Public Roads of the United States Department of Agriculture.[1] In general, they may be accepted as standard procedures for use in the examination of bituminous road materials.

1. Classification of Commonly Used Bituminous Materials. (A) NATURAL. (1) *Asphalt or "Mineral Pitch."*[2]—Trinidad, Venezuelan, Californian, and other native asphalts may contain small or large percentages of extraneous mineral matter (calcareous, silicious or earthy matrix) and water.

(2) *Gilsonite or Uintaite.*—An asphaltite which is nearly pure bitumen.[3] Glance pitch (manjak) and grahamite are other asphaltites.

[1] *Bulletin* **314** (1915), 48 pp., by P. HUBBARD and C. S. REEVE; *cf.* the *Appendix*, pages 385 *et seq.*.

[2] See RICHARDSON, *J. Soc. Chem. Ind.*, **17** (1898), 13.

[3] DAY, *J. Frank. Inst.*, **140** (1895), 221; *Proc. Am. Phil. Soc.*, **37**, 171.

Related minerals are the asphaltic pyrobitumens, elaterite, wurtzilite, albertite, and impsonite. These and the other bitumens are described in Abraham's "Asphalts and Allied Substances."

(3) *Asphaltic Limestones and Sands.*—The proportion of bitumen ("tar") usually is not over 12 to 15 per cent.[1]

(4) *Maltha.*—A viscous hydrocarbon complex, also known as *pissasphalt.*

(B) ARTIFICIAL. (1) *Straight Petroleum Asphalt.*—A residue left after the sufficient distillation of petroleum; it is practically pure bitumen. The pyrogenous treatment of wurtzilite also gives an artificial asphalt.

(2) *Oxidized or Blown* (Byerly and Culmer processes) and *Sulphurized* (Dubbs process) *Residues.*[2]—These products are essentially pure bitumens.

(3) *Tars and Tar Products.*—Nearly pure bitumens.

(4) *Emulsions.*—These may contain foreign material, such as water and salts of organic acids.

2. Scheme of Examination.—All petroleum, maltha, and solid native bitumen products are subjected to the following tests: Specific gravity; volatilization at 163°C.; bitumen soluble in carbon disulphide; bitumen insoluble in 86°Bé. paraffin naphtha; and fixed carbon.

Of these types the very fluid and sometimes the more viscous products may be submitted to viscosity, flash-, and burning-point determinations. Very viscous materials, too soft for the penetration test, are subjected to the float test, and semi-solid and solid products to the penetration test. If the material is sufficiently hard at ordinary temperatures, a melting-point determination also may prove of value. Sometimes two or more of the above mentioned tests, depending upon the character of the material and the use to which it is to be put, may be made to advantage on a single material.[3] When, for any reason, it is suspected that the material under examination has been overheated and possibly injured during process of manufacture, or prepared from a solid

[1] See *Rept.* **281,** Mines Branch, Canada Dept. of Mines; and also the following *Summary Reports,* Mines Branch, Canada Dept. of Mines: **1913,** 54; **1914,** 62; **1915,** 68; **1916,** 56; and **1918,** 194.

[2] See BACON and HAMOR, *lib. cit.,* **2,** 620 and 626.

[3] On distinguishing between natural and artificial asphalts, see GRAEFE, *Z. angew. Chem.,* **29** (1916), i, 21. See also PAILLER, *Caoutchouc & gutta percha,* **16** (1919), 10063.

native indurated bitumen, the determination of bitumen insoluble in carbon tetrachloride may be made. The paraffin scale determination is made on those materials which are to be identified as being partly composed of heavy paraffin hydrocarbons. The residue obtained from the volatilization test usually is subjected to either the float or penetration test, and, in addition, it may be examined according to any or all of the above-mentioned tests, as occasion may require.

Tar and tar products are submitted to the following tests: Specific gravity; distillation; and bitumen soluble in carbon disulphide.

Petroleum and asphalt emulsions are subjected to some of the methods of examination applicable to fluid and viscous residual petroleums and also to the following analytical determinations: water, ammonia, fixed alkali, and fatty and resin acids.

In addition, the viscosity test may be employed for fluid products, and it is usually desirable that the float test be made on all the viscous and semi-solid tar products. Melting-point determinations also are made on the more or less solid refined tars or tar pitches. Mixtures of tar and petroleum or asphalt products are, in addition, subjected to the dimethyl sulphate test.

Some exceptional materials cannot be examined satisfactorily according to any one predetermined scheme, and at the present time this matter must be left to the judgment and experience of the analyst. Practically all of the methods described hereinafter are, however, applicable to the more common materials, and for a given material those methods should be selected which will give the most information concerning its character and suitability for the specific use for which it is intended.

Bituminous aggregates are examined first for the percentage of bitumen soluble in carbon disulphide. If the amount is in excess of 5 per cent., an extraction is made on a large sample and the recovered bitumen is examined according to one of the previously mentioned schemes if it can be identified, or, if not, it is submitted to those tests which are of most value, as suggested above. The extracted mineral aggregate usually is graded quantitatively, and, if it is to be used or has been used as an integral part of the road proper, its percentage of voids sometimes is determined.

Forms for reporting the results of the examination of bitumin-

ous road materials according to the methods described herein, are given in the *Appendix*, page 383.

3. Specific Gravity.—(*A*) PYCNOMETER METHOD (used for viscous fluid and semi-solid bitumens and emulsions).

Apparatus.—1 large metal kitchen spoon; 1 steel spatula or kitchen knife; 1 bunsen burner and rubber tubing; 1 250-c.c. low-form glass beaker; 1 chemical thermometer reading from −10°C. to 110°C.; 1 special pycnometer (Fig. 53); and 1 analytical balance, capacity 100 g., sensitive to 0.1 mg.

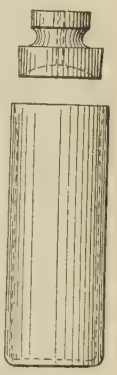

FIG. 53.—Specific gravity bottle (Hubbard type).

Procedure.—The inconvenience and difficulty of employing the ordinary narrow-neck pycnometer, when determining the specific gravity of viscous fluid and semi-solid bitumens, has led to the use of the special form shown in Fig. 53.

This pycnometer consists of a fairly heavy, straight-walled glass tube, 70 mm. long and 22 mm. in diameter, carefully ground to receive an accurately fitting solid glass stopper with a hole of 1.6 mm. bore, in place of the usual capillary opening. The lower part of this stopper is made concave in order to allow all air bubbles to escape through the bore. The depth of the cup-shaped depression is 4.8 mm. at the center. The stoppered tube has a capacity of about 24 c.c. and when empty weighs about 28 g. Its principal advantages are (1) that any desired amount of bitumen may be poured in without touching the sides above the level desired; (2) it is easily cleaned; and (3), on account of the 1.6 mm. bore, the stopper can be more easily inserted when the tube is filled with a very viscous oil than if it contained a capillary opening.

When working with semi-solid bitumens which are too soft to be broken and handled in fragments, the following method of determining specific gravity is employed. The clean, dry pycnometer first is weighed empty and this weight is called "*a*." It then is filled in the usual manner with freshly distilled water at 25°C., and the weight again is taken and called "*b*." A small amount of the bitumen should be placed in the spoon and brought to a fluid condition by the gentle application of heat, with care that no loss by evaporation occurs. When sufficiently fluid, enough is poured into the dry pycnometer, which also may be

warmed, to fill it about half full, without allowing the material to touch the sides of the tube above the desired level. The tube and contents then are allowed to cool to room temperature, after which the tube with the stopper is weighed. This weight is called "*c*." Distilled water, at 25°C., then is poured in until the pycnometer is full. After this, the stopper is inserted, and the whole is cooled to 25°C. by a 30-minute immersion in a beaker of distilled water maintained at that temperature. All surplus moisture then is removed with a soft cloth, and the pycnometer and contents are weighed. This weight is called "*d*." From the weights obtained the specific gravity of the bitumen may be calculated readily by the following formula: Specific gravity at 25°C./25°C. $= \frac{c-a}{(b-a)-(d-c)}$.

Both "*a*" and "*b*" are constants and need be determined but once. It is therefore necessary to make but two weighings for each determination after the first. Results obtained according to the method given *supra* are accurate to within 2 units in the third decimal place, while the open-tube method is accurate to the second decimal place only.

The specific gravity of fluid bitumens may be determined in the ordinary manner with the pycnometer by completely filling it with the material and dividing the weight of the bitumen thus obtained by that of the same volume of water.

The pycnometer may be cleaned by placing it in a hot-air bath until the bitumen is sufficiently fluid to pour. As much is drained out as possible and the interior is swabbed with a piece of cotton waste. It then is rinsed clean with a little carbon disulphide and, after drying, is again ready for use.

(B) Displacement Method (used for hard solid bitumens).

Apparatus.—1 chemical thermometer reading from −10°C. to 110°C.; 1 analytical balance, capacity 100 g., sensitive to 0.1 mg.; 1 wood or metal platform; 1 150-c.c. low-form glass beaker; and 1 piece of fine silk thread.

Procedure.—For materials which are hard enough to be broken and handled in fragments at room temperature, the following method will prove convenient. A small fragment of the bitumen (about 1 c.c.) is suspended by means of a silk thread from the hook on one of the pan supports, about 1½ in. above the pan, and weighed. This weight is called "*a*." It then is weighed when immersed in water at a temperature of 25°C., as shown in

Fig. 54, and this weight is called "*b*." The specific gravity then may be calculated by means of the following formula:

$$\text{Specific gravity} = \frac{a}{a-b}$$

USE OF SPECIFIC GRAVITY DETERMINATION.—The specific gravity determination is made on all bitumens containing less than 50 per cent. of mineral matter, and also on bitumens recovered from bituminous aggregates. The specific gravity usually is reported to the third decimal place.

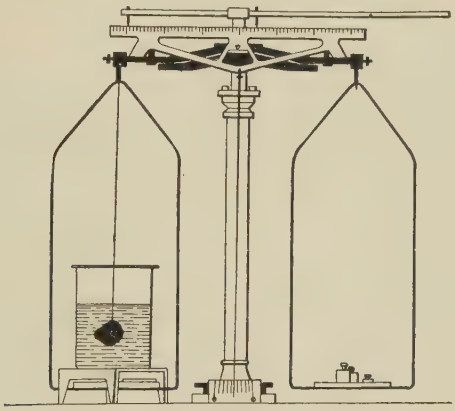

FIG. 54.—An illustration of the displacement method of determining specific gravity.

4. Float Test. *Apparatus.*—1 aluminum float or saucer; 2 conical brass collars; 2 1-quart tin cups, seamless; 2 chemical thermometers reading from −10°C. to 110°C.; 1 iron tripod; 1 bunsen burner and rubber tubing; 1 burette clamp and support; 1 large metal kitchen spoon; 1 steel spatula or kitchen knife; 1 brass plate, 5 by 8 cm.; and 1 stop-watch.

Procedure.—The float apparatus (Fig. 55) consists of two parts, an aluminum float or saucer and a conical brass collar. The two parts are made separately, so that one float may be used with a number of brass collars.

In making the test the brass collar is placed with the small end down on the brass plate, which previously has been amalgamated with mercury by first rubbing with a dilute solution of mercuric chloride or nitrate and then with mercury. A small quantity of the material to be tested is heated in the metal spoon until quite

fluid, with care that it undergoes no appreciable loss by volatilization and that it is kept free from air bubbles. It then is poured into the collar in a thin stream until slightly more than level with the top. The surplus may be removed, after the material has cooled to room temperature, by means of a spatula or steel knife which has been heated slightly. The collar and plate then are placed in one of the tin cups containing ice water maintained at 5°C., and are left in this bath for at least 15 minutes. Meanwhile the other cup is filled about three-fourths full of water and placed on the tripod, and the water is heated to any desired

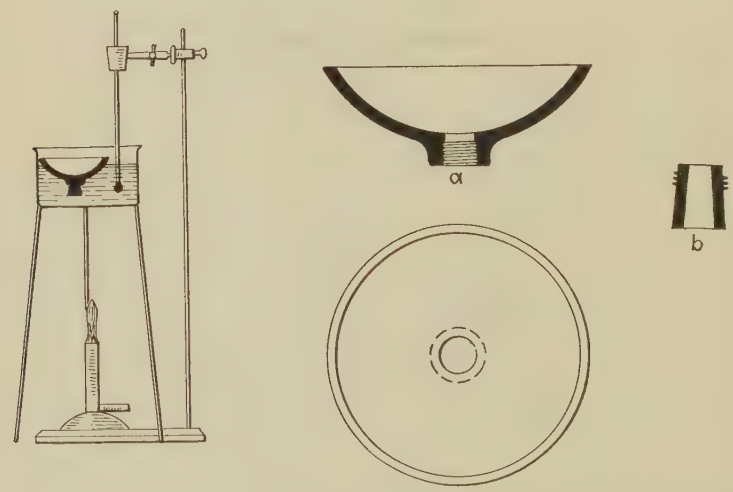

FIG. 55.—Apparatus for use in carrying out the float test (New York Testing Laboratory apparatus): *a*, aluminum float or saucer; *b*, conical brass collar.

temperature at which the test is to be made. This temperature should be maintained accurately and should at no time throughout the entire test be allowed to vary more than 0.5°C. from the temperature selected. After the material to be tested has been kept in the ice water for at least 15 minutes, the collar with its contents is removed from the plate and screwed into the aluminum float, which then is floated immediately in the warmed bath. As the plug of bituminous material becomes warm and fluid, it is forced gradually upward and out of the collar, until water gains entrance to the saucer and causes it to sink.

The time in seconds between placing the apparatus on the water and when the water breaks through the bitumen, is deter-

mined by means of a stop-watch and is taken as a measure of the consistency of the material under examination.

Use of the Float Test.—This test always is made on viscous and semi-solid refined tars, and often on the viscous and semi-solid petroleum and asphalt products, although, when the penetration test can be employed on the two latter classes of material, the float test is not always considered necessary. For the more fluid products the test is carried out at 32°C.; and for the semi-solid materials, at 50°C. When the material under examination is quite hard, the test may be run at 100°C.

The float test is a most convenient one for roughly checking the uniformity of different shipments of bituminous material furnished under specifications.

5. Penetration Test. *Apparatus.*—1 penetrometer complete, with a seconds pendulum or metronome; 1 tin box, approximately 5 cm. in diameter by 3.5 cm. in height; 1 large metal kitchen spoon; 1 steel spatula or kitchen knife; 1 glass penetration dish, approximately 10 cm. in diameter by 6 cm. high; 1 enamel-ware dish, approximately 3 in. deep and 9 in. in diameter; and 1 chemical thermometer reading from -10° to 110°C.

Procedure.—The object of the penetration test is to ascertain the consistency of the material under examination by determining the distance a weighted needle will penetrate it at a given temperature. A standard needle is employed for this purpose and this needle usually is weighted with 100 g. The depth of penetration is determined upon the bitumen maintained at 25°C., while the load is applied for five seconds.

The standard needle is made from round, polished, annealed-steel drill rod having a diameter of from 0.0405 to 0.0410 in. The rod is tapered to a sharp point at one end, with the taper extending back ¼ in. It then is polished highly, tempered, and again polished with jeweler's rouge. The finished needle is from 1¾ to 2 in. in length and exactly 0.040 in. in diameter. This needle, as made in the laboratory by Hubbard and Reeve, gives the same results as the old standard No. 2 cambric needle and possesses the advantage that it can be duplicated exactly and described accurately.

The penetration apparatus shown in Fig. 56 consists of a standard needle "*a*," inserted in a short brass rod, which is held in the aluminum rod "*b*" by a binding screw. The aluminum rod is secured in a framework so weighted and balanced that,

when it is supported on the point of the needle, the framework and rod will stand in an upright position, allowing the needle to penetrate perpendicularly without the aid of a support.

The frame, aluminum rod, and needle weigh 100 g. with the weight "*c*" on the bottom of the frame, while without the weight they weigh 50 g. Fig. 56 shows the needle and weighted frame, together with side and front views of the entire apparatus, put together and ready for making a penetration. The shelf for the sample is marked "*d*;" "*e*" is the clamp to hold the aluminum

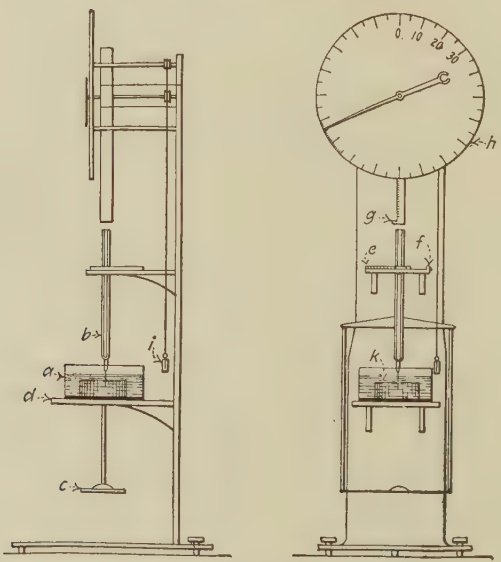

Fig. 56.—Dow penetration machine.

rod until it is desired to make a test; and "*f*" is a button which, when pressed, opens the clamp. By turning this button while the clamp is being held open, it will lock and keep the clamp from closing until unlocked. The device for measuring the distance penetrated by the needle consists of a rack, with a foot "*g*." The movement of this rack turns a pinion, to which is attached the hand which indicates on the dial "*h*" the vertical distance covered by the rack. One division of the dial corresponds to a movement of 0.1 mm. by the rack. The rack may be raised or lowered by moving the counterweight "*i*" up or down. The tin box containing the sample to be tested is marked

"*k*;" this is submerged in water contained in the glass cup, in order to maintain a constant temperature.

This apparatus is known as the Dow penetration machine. Another type of machine, known as the New York Testing Laboratory penetrometer, based upon the same general principle and using the same standards, is at present employed by the Office of Public Roads and Rural Engineering. This penetrometer is shown in Fig. 57. Both machines give practically the same results, if operated under the same conditions, and it is therefore unnecessary to include a description of the last-mentioned instrument.

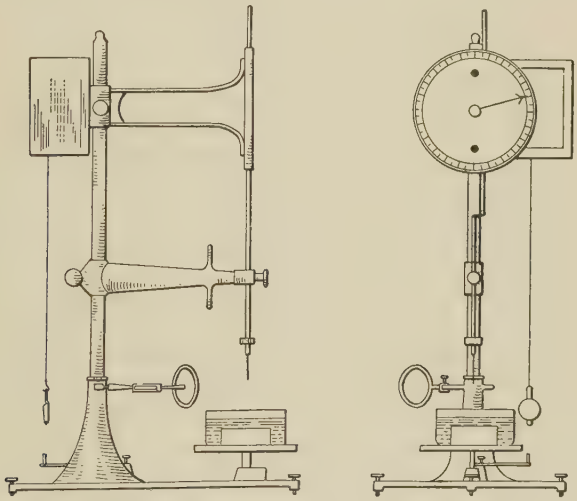

FIG. 57.—New York Testing Laboratory penetrometer.

A cup suitable for holding the box containing the test sample during penetration is made conveniently from a glass crystallizing dish, 10 cm. in diameter, with straight sides about 6 cm. high. Three right triangles, with right-angle sides 1 and 5 cm., respectively, are cut from $\frac{1}{16}$ in. sheet metal. Some solid bitumen is melted in the bottom of the dish, forming a layer about $\frac{1}{8}$-in. thick, into which the triangles are placed, resting on the side 5 cm. long. Their apexes should meet at the center, with their short sides dividing the circumference of the dish into three equal arcs. When the bitumen has hardened, the triangles give a firm support for circular boxes, and the possiblity of any rocking motion and consequent faulty results is avoided.

The penetration test is conducted as follows.[1] A sample of the material to be tested is warmed sufficiently to flow, and poured into the tin box. The box and contents, after cooling for 30 minutes at room temperature, are immersed in water maintained at the temperature at which the test is to be made, and allowed to remain immersed for one and one-half hours. The sample in the tin box now should be placed in the glass cup and removed in it, covered with as much water as convenient without spilling, to the shelf "*d*." The brass rod with the needle is inserted into "*b*" and secured by tightening the binding screw. The rod is lowered until the point of the needle almost touches the surface of the sample; then, by grasping the frame with both hands, it is pulled down cautiously until the needle just comes in contact with the surface of the sample. This can be seen best by having a light so situated that, upon looking through the sides of the glass cup, the needle will be reflected from the surface of the sample. After thus setting the needle, the counterweight is raised slowly until the foot of the rack rests on the head of the rod and a reading of the dial is taken. The clamp then is opened wide by pressing the button and is held in this position for exactly five seconds, as determined by the pendulum or metronome. The clamp then is released, the rack lowered until it rests on the rod, and the difference between the first and second readings of the dial in hundredths of a centimeter is taken as the distance penetrated by the needle.

Owing to the susceptibility of certain bitumens to slight changes in temperature, the water-bath should be maintained accurately at the desired temperature, both before and during the test, and, when the room temperature differs greatly from that of the bath, the water in the glass cup should be renewed after each test. An average of from three to five tests, which should not differ more than four points between maximum and minimum, is taken as the penetration of the sample. The tests should be made at points on the surface of the sample not less than 1 cm. from the side of the container and not less than 1 cm. apart.

The needle should be removed and cleaned thoroughly by

[1] On the standard test for penetration of bituminous materials issued by the American Society for Testing Materials, see "A. S. T. M. Standards," **1918,** 632. Newton and Williams (*Pet. Age*, **6** (1919), 241) recommend the Humbolt penetrometer.

wiping with a dry cloth, after which it is ready for another test. The point of the needle should be examined from time to time with a magnifying glass, to see that it is not injured in any way. If it is found defective, it may be removed by heating the brass rod and withdrawing with pliers. A new needle then may be inserted in the heated brass rod, and held firmly in place by a drop of soft solder.

USE OF THE PENETRATION TEST.—This test is made on all semi-solid and solid oil-asphalts, asphaltic cements, and native asphalts, but seldom on tar products. It is also often made on the residues of materials subjected to the volatilization test, when they are sufficiently hard. For work on residues, which seldom amount to more than 20 c.c., a small container, which should not, however, be less than 1 in. in diameter, will be required.

While the standard conditions under which this test is made call for a 100-g. load applied for five seconds on the material maintained at a temperature of 25°C., it is sometimes desirable, when very soft materials are tested, to make the test with a 50-g. weight. In order to ascertain how susceptible a material may be to temperature changes, tests may be made at any other desired temperatures, preferably 0°C., with a 200-g. weight for one minute and at 46°C. with a 50-g. weight for five seconds.

In all cases the results of tests should be reported in hundredths of a centimeter, as follows, showing all the conditions in order that no misinterpretation of results may occur:

Penetration (.... seconds, ... grams at ....°C.) = ...

6. Melting-Point.[1] BALL AND RING METHOD.—The *apparatus* consists of a brass ring, $\frac{1}{4}$ in. deep and $\frac{5}{8}$ in. in inside diameter, with walls $\frac{3}{32}$ in. thick, fastened at right angles to a wire; and a polished steel ball, $\frac{3}{8}$ in. in diameter, and weighing between 3.45 and 3.5 g. (see Fig. 133 in the *Appendix*).

[1] A comparison of methods for determining the melting-points of asphalts (*Chem. Met. Eng.*, **21** (1919), 81) indicates that the Richardson ball method gives very close checks, but has the disadvantage that personal error enters largely into the manipulation. The General Electric method gives fair checks, but is unsuited for factory control. The Kramer and Sarnow method gives rather the poorest checks and is more or less complicated and tedious. The ball and ring method adopted by the American Society for Testing Materials is well adapted to specifications, but is not very well suited for factory control work, since but one sample can be tested at a time (see page 386).

Procedure.—A small quantity of asphalt is melted in a tablespoon and poured into the ring, which is set on an amalgamated brass plate, allowing for shrinkage. When it is cool, the excess is cut off with a hot knife and the ball set on the asphalt in the ring. The ring is inserted in one hole of a two-hole cork and through the other a thermometer is inserted so that the ball is at the same level as the ring, and the whole is suspended in a water bath, 1 in. above the bottom of the beaker. Heat is applied in sufficient quantity to raise the temperature at the rate of 9°F. per minute. As it is heated the ball gradually sinks into the asphalt, going through and forming a pocket with the ball

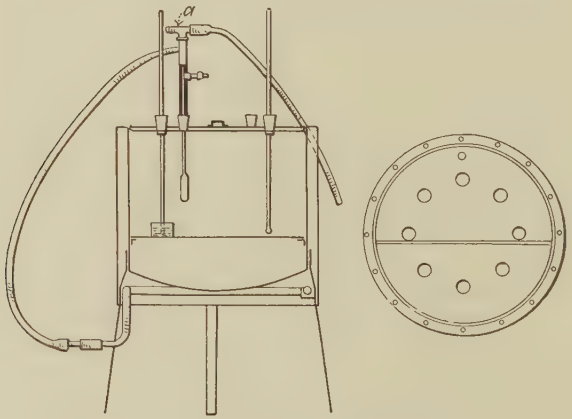

FIG. 58.—New York Testing Laboratory oven.

inside. The temperature at which the pocket touches the bottom of the beaker is taken as the melting-point.

For test of 110°F. (43.3°C.) melting-point pitch, the sample should be cooled to 50°F. (10°C.); for 210°F. (98.8°C.), to 100°F. (37.7°C.).

7. Volatilization Test. *Apparatus.*—1 constant-temperature hot-air oven with rubber tubing (Fig. 58); 1 thermo-regulator; 2 chemical thermometers reading from −10° to 250°C.; 1 tin box, 6 cm. in diameter by 2 cm. deep; and 1 analytical balance, capacity 100 g., sensitive to 0.1 mg.

Procedure.—The object of the volatilization test is to determine the percentage of loss which the material undergoes when 20 g. in a standardized container are subjected to a uniform tem-

perature of 163°C. for five hours, and also to ascertain any changes in the character of the material due to such heating.

The oven shown in Fig. 58, known as the New York Testing Laboratory oven, is used by the Office of Public Roads and Rural Engineering, although any other form may be used that will give a uniform temperature throughout all parts where samples are placed. The bulb of one of the thermometers is immersed in a sample of some fluid, non-volatile bitumen, while the other is kept in air at the same level. The first thermometer serves to show the temperature of the samples during the test, while the latter gives prompt warning of any sudden changes in temperature due to irregularities in the gas pressure, etc.

Before making the test the interior of the oven should show a temperature of 163°C., as registered by the thermometer in air. The tin box is weighed accurately after carefully wiping with a towel to remove any grease or dirt. About 20 g. of the material to be tested then are placed in the box. The material then may be weighed on a rough balance, if one is at hand, after which the accurate weight, which should not vary more than 0.2 g. from the specified amount, is obtained. It may be necessary to warm some of the material in order to handle it conveniently, after which it must be allowed to cool before determining the accurate weight.

The sample now should be placed in the oven, where it is allowed to remain for a period of five hours, during which time the temperature as shown by the thermometer in bitumen should not vary at any time more than 2°C. from 163°C. The sample then is removed from the oven, allowed to cool, and reweighed. From the difference between this weight and the total weight before heating, the percentage of loss on the amount of material taken is calculated.

The general appearance of the residue should be noted, especially with regard to any changes which the material may have undergone. Some relative idea of the amount of hardening which has taken place may be obtained from the results of a float or penetration test made on the residue, as compared with the results of the same test on the original sample. It is also frequently desirable to make the specific gravity and other tests on the residue for the purpose of identifying or ascertaining the character of the base used in the preparation of cut-back products. Before any tests are made on the residue, it should be melted and stirred thoroughly while cooling.

Highly volatile and non-volatile materials should not be subjected to this test at the same time in the same oven, owing to a tendency on the part of the latter to absorb some of the volatile products of the former.

Use of the Volatilization Test.—The volatilization test, as described above, is made on practically all bitumens with the exception of tars, for which the distillation test answers a similar purpose. The test also is made frequently at 105°C. for five hours, and with products containing small amounts of water it is usually necessary to make a test at the lower temperature before the material can be heated at 163°C. without foaming over.[1] In the case of emulsions it is customary to determine the loss on a 20-g. sample at room temperature for 24 hours, after which the sample is heated at 105°C. for five hours. This additional loss is obtained and all determinations are made on the dried residue and reported accordingly.

The volatilization test occasionally is made at 205°C. for five hours on a fresh sample, in order to show the effect of this higher temperature as compared with the results at 163°C.

Because of the fact that, after the volatilization test, it frequently happens that a penetration test cannot be made upon the residue of a 20-g. sample in the container specified, it has been suggested that the volatilization test be made upon a 50-g. sample in a tin box, 5½ cm. in diameter and 3½ cm. in depth. In many cases, however, the percentage loss by volatilization and the consequent hardening will be found to vary materially from that obtained with a 20-g. sample, owing to differences in the ratio of exposed surface area to total volume of material. This fact should be borne in mind if the test is made with a 50-g. sample.

8. Bitumen Soluble in Carbon Disulphide (Total Bitumen). *Apparatus.*—1 100-c.c. Erlenmeyer flask; 1 500-c.c. flask with side-neck, for filtering under pressure; 1 rubber stopper with one hole; 1 filter-tube, 3.9 cm. inside diameter; 1 platinum or porcelain Gooch crucible; 1 piece of seamless rubber tubing, about 3 cm. in diameter and 3 cm. long; 50 g. of long-fiber amphibole asbestos; 2 wash-bottles, 1 for solvent and 1 for water; 1 bunsen burner; 1 nichrome triangle; 1 iron tripod; 1 drying oven; 1

[1] For the standard test for loss on heating of oil and asphaltic compounds issued by the American Society for Testing Materials, see "A. S. T. M. Standards," **1918,** 635.

desiccator with calcium chloride; 1 thermometer reading from −10° to 110°C.; 1 vacuum pump and connections; and 1 analytical balance, capacity 100 g., sensitive to 0.1 mg.

Procedure.—This test consists in dissolving the bitumen in carbon disulphide and recovering any insoluble matter by filtering the solution through an asbestos felt. The form of Gooch crucible best adapted for the determination is 4.4 cm. wide at the top, tapering to 3.6 cm. at the bottom, and is 2.5 cm. deep

For preparing the felt the necessary apparatus is arranged as shown in Fig. 59, in which "*a*" is the filtering flask, "*b*" a rubber stopper, "*c*" the filter-tube, and "*d*" a section of rubber tubing which tightly clasps the Gooch crucible "*e*." The asbestos is cut with scissors into pieces not exceeding 1 cm. in length, after which it is shaken up with just sufficient water to pour easily. The crucible is filled with the suspended asbestos, which is allowed to settle for a few moments. A light suction then is applied to draw off all the water and leave a firm mat of asbestos in the crucible. More of the suspended material is added, and the operation is repeated until the felt is so dense that it scarcely transmits light when held so that the bottom of the crucible is between the eye and the source of light. The felt then should be washed several times with water, and drawn firmly against the bottom of the crucible by an increased suction. The crucible is removed to a drying oven for a few minutes, after which it is ignited at red heat over a bunsen burner, cooled in a desiccator and weighed.

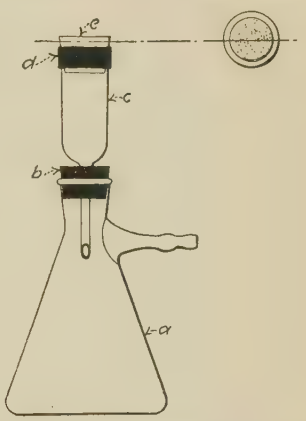

FIG. 59.—Apparatus for determining soluble bitumen.

From 1 to 2 g. of bitumen, or about 10 g. of an asphalt topping or rock asphalt, now are placed in the Erlenmeyer flask, which previously has been weighed, and the accurate weight of the sample is obtained. 100 c.c. of chemically pure carbon disulphide are poured into the flask in small portions, with continual agitation, until all lumps disappear and nothing adheres to the bottom. Then the flask is corked and set aside for 15 minutes.

After being weighed, the Gooch crucible containing the felt is

set up over the dry pressure flask, as shown in Fig. 59, and the solution of bitumen in carbon disulphide is decanted through the felt without suction by gradually tilting the flask, with care not to stir up any precipitate that may have settled out. At the first sign of any sediment coming out, the decantation is stopped and the filter is allowed to drain. A small amount of carbon disulphide then is washed down the sides of the flask, after which the precipitate is brought upon the felt and the flask scrubbed, if necessary, with a "policeman," to remove all adhering material. The contents of the crucible are washed with carbon disulphide, until the washings run colorless. Suction then is applied until there is practically no odor of carbon disulphide in the crucible, after which the outside of the crucible is cleaned with a cloth moistened with a small amount of the solvent. The crucible and contents are dried in the hot-air oven at 100°C. for about 20 minutes, cooled in a desiccator, and weighed. If any appreciable amount of insoluble matter adheres to the flask, it also should be dried and weighed, and any increase over the original weight of the flask should be added to the weight of insoluble matter in the crucible. The total weight of insoluble material may include both organic and mineral matter. The former, if present, is burned off by ignition at a red heat until no incandescent particles remain, thus leaving the mineral matter or ash, which can be weighed on cooling. The difference between the total weight of material insoluble in carbon disulphide and the weight of substance taken, equals the total bitumen, and the percentage weights are calculated and reported as total bitumen, and organic and inorganic matter insoluble, on the basis of the weight of material taken for analysis.

This method is quite satisfactory for straight oil and tar products, but where certain natural asphalts are present it will be found practically impossible to retain all the finely divided mineral matter on an asbestos felt. It is, therefore, generally more accurate to obtain the result for total mineral matter by direct ignition of a 1-g. sample in a platinum crucible or to use the result for ash obtained in the fixed carbon test. The total bitumen then is determined by deducting from 100 per cent. the sum of the percentages of total mineral matter and organic matter insoluble. If the presence of a carbonate mineral is suspected, the percentage of mineral matter may be obtained most accurately by treating the ash from the fixed carbon determina-

tion with a few drops of ammonium carbonate solution, drying at 100°C., then heating for a few minutes at a dull red heat, cooling, and weighing again.

When difficulty in filtering is experienced—for instance, when Trinidad asphalt is present in any amount—a period of longer subsidence than 15 minutes is necessary, and the following method proposed by the Committee on Standard Tests for Road Materials of the American Society for Testing Materials is recommended:[1]

After drying, from 2 to 15 g. (depending on the richness in bitumen of the substance) are weighed into a 150-c.c. Erlenmeyer flask, the tare of which has been ascertained previously, and treated with 100 c.c. of carbon disulphide. The flask is then loosely corked and shaken from time to time until practically all large particles of the material have been broken up, when it is set aside and not disturbed for 48 hours. The solution is then decanted off into a similar flask that has been previously weighed, as much of the solvent being poured off as possible without disturbing the residue. The first flask is again treated with fresh carbon disulphide and shaken as before, when it is put away with the second flask and not disturbed for 48 hours.

At the end of this time the contents of the two flasks are carefully decanted off upon a weighed Gooch crucible fitted with an asbestos filter, the contents of the second flask being passed through the filter first. The asbestos filter shall be made of ignited long-fiber amphibole, packed in the bottom of a Gooch crucible to the depth of not over one-eighth of an inch. After passing the contents of both flasks through the filter, the two residues are shaken with more fresh carbon disulphide and set aside for 24 hours without disturbing, or until it is seen that a good subsidation has taken place, when the solvent is again decanted off upon the filter. This washing is continued until the filtrate or washings are practically colorless.

The crucible and both flasks are then dried at 125°C. and weighed. The filtrate containing the bitumen is evaporated, the bituminous residue burned, and the weight of the ash thus obtained added to that of the residue in the two flasks and the crucible. The sum of these weights deducted from the weight of substance taken gives the weight of bitumen extracted.

Use of Total Bitumen Determination.—This determination is made on all classes of bituminous products. In the

[1] *Proc. Am. Soc. Testing Materials*, **9** (1909), 221; see also "A. S. T. M. Standards," **1918**, 630.

analysis of tars the organic matter insoluble is commonly known and reported as "free carbon."

The method described by Richardson[1] is to be used in case the material under examination contains appreciable quantities of mineral matter. The procedure is the same as that described *supra*, except that the carbon disulphide which has passed the filter is permitted to subside for twenty-four hours and is decanted carefully from the flask in which it has been received into a weighed platinum or unweighed porcelain dish. If there is any sediment in the flask it must be rinsed back into the Gooch crucible with carbon disulphide and the crucible again washed clean. The solvent in the dish is placed in a good draught and lighted. When all the disulphide has burned, the bitumen remaining in the dish is burned off over a lamp and the mineral residue, which was too fine to subside, is weighed if the burning was done in a platinum dish, or dusted out and added to the crucible if in a porcelain one. In the former case the weight is added to that of the Gooch crucible or subtracted from the percentage of bitumen found without its consideration, as a correction. Care must be taken in this method of procedure that the solvent does not creep over the sides of the crucible and that the outside is free from bitumen before weighing.

9. Determination of Bitumen Insoluble in Paraffin Naphtha. *Apparatus.*—The apparatus is the same as for the determination of bitumen soluble in carbon disulphide.

Procedure.—This determination is made in the same general manner as the total bitumen determination, except that 100 c.c. of 86° to 88°Bé. paraffin naphtha, at least 85 per cent. distilling between 40°C. and 95°C., are employed as solvent instead of carbon disulphide. Considerable difficulty sometimes is experienced in breaking up some of the heavy semi-solid bitumens; the surface of the material is attacked, but it is necessary to remove some of the insoluble matter in order to expose fresh material to the action of the solvent. It is, therefore, advisable to heat the sample after it is weighed, allowing it to cool in a thin layer around the lower part of the flask. If difficulty still is experienced in dissolving the material, a rounded glass rod will be found convenient for breaking up the undissolved particles. Not more than one-half of the total amount of naphtha required should be used until the sample entirely is broken up. The

[1] "The Modern Asphalt Pavement," 2nd ed.

balance of the 100 c.c. then is added, and the flask is twirled a moment in order to mix the contents thoroughly, after which it is corked and set aside for 30 minutes.

In making the filtration the utmost care should be exercised to avoid stirring up any of the precipitate, in order that the filter may not be clogged and that the first decantation may be as complete as possible. The sides of the flask then should be washed down quickly with naphtha and, when the crucible has drained, the bulk of insoluble matter is brought upon the felt. Suction may be applied when the filtration by gravity almost ceases, but should be used sparingly, as it tends to clog the filter by packing the precipitate too tightly. The material on the felt never should be allowed to run entirely dry until the washing is completed, as shown by the colorless filtrate. When considerable insoluble matter adheres to the flask, no attempt should be made to remove it completely. In such cases the adhering material is washed until free from soluble matter, and the flask is dried with the crucible at 100°C. for about one hour, after which it is cooled and weighed. The percentage of bitumen insoluble is reported upon the basis of total bitumen taken as 100.

The difference between the material insoluble in carbon disulphide and in the naphtha is the bitumen insoluble in the latter. Thus, if in a certain instance it is found that the material insoluble in carbon disulphide amounts to 1 per cent. and that 10.9 per cent. is insoluble in naphtha, the percentage of bitumen insoluble would be calculated as follows:

$$\frac{\text{Bitumen insoluble in naphtha}}{\text{Total bitumen}} = \frac{10.9 - 1}{100 - 1} = \frac{9.9}{99} = 10 \text{ per cent.}$$

Use of the Naphtha-Insoluble Bitumen Determination. —This test is made on all petroleums, malthas, and asphalts and other solid native bitumens, and their products.

It should be noted that petroleum naphthas are by no means definite compounds, but are composed of a number of hydrocarbons which vary in character and quantity according to the petroleum from which they have been distilled. Their solvent powers also vary greatly. Thus naphthas produced from asphaltic petroleums, consisting mainly of naphthene and polymethylene hydrocarbons, are much more powerful solvents of the heavier asphaltic hydrocarbons than are the paraffin naphthas. The density of the naphtha also affects its solvent power, for heavy naphthas dissolve the heavier hydrocarbons more readily

than do the lighter naphthas. As the main object of this test is to separate the heavier hydrocarbons of an asphaltic nature from the paraffin hydrocarbons, a paraffin solvent should be employed, and for ordinary purposes a paraffin naphtha of 86° to 88°Bé. as described has been found to be readily obtainable and fairly satisfactory.

The determination also is made with heavier naphthas, such as 66°Bé. and 72°Bé., for the purpose of grading the character of the bitumen present in the compound. Therefore a report always should state distinctly the gravity and character of the solvent used.

10. Determination of Fixed Carbon. *Apparatus.*—1 iron ring-support (ring, 7.5 cm. in diameter); 1 platinum or nichrome triangle; 1 bunsen burner and rubber tubing; 1 platinum crucible with a tight-fitting cover (weight complete, from 20 to 30 g.); 1 crucible tongs; 1 desiccator with calcium chloride; and 1 analytical balance, capacity 100 g., sensitive to 0.1 mg.

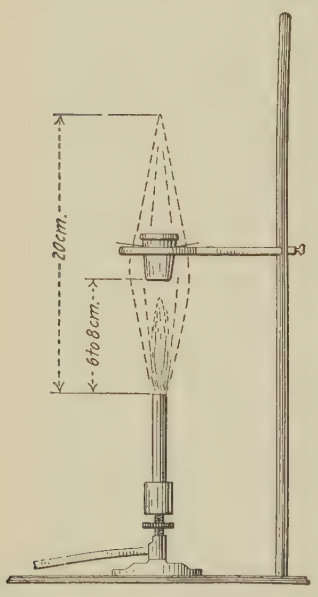

FIG. 60. Apparatus for the determination of fixed carbon.

Procedure.—This determination is made in accordance with the method described for coal in the *Journal of the American Chemical Society*, **21** (1899), 1116. One g. of the material is placed in a platinum crucible weighing from 20 to 30 g. and having a tightly fitting cover. It then is heated for seven minutes over the full flame of a bunsen burner, as shown in Fig. 60. The crucible should be supported on a platinum triangle with the bottom from 6 to 8 cm. above the top of the burner. The flame should be fully 20 cm. high when burning freely, and the determination should be made in a place free from drafts. The upper surface of the cover should burn clear, but the under surface should remain covered with carbon, excepting in the case of some of the more fluid bitumens, when the under surface of the cover may be quite clean.

The crucible is removed to a desiccator and, when cool, is weighed, after which the cover is removed, and the crucible is placed in an inclined position over the bunsen burner and ignited until nothing but ash remains. Any carbon deposited on the cover also is burned off. The weight of ash remaining is deducted from the weight of the residue after the first ignition of the sample. This gives the weight of the so-called fixed or residual carbon, which is calculated on a basis of the total weight of the sample exclusive of mineral matter. If the presence of a carbonate mineral is suspected, the percentage of mineral matter may be obtained most accurately by treating the ash with a few drops of ammonium carbonate solution, drying at 100°C., then heating for a few minutes at a dull red heat, cooling and weighing.

An excellent form of crucible for this test is shown in Fig. 60. It has a cover with a flange 4 mm. wide, fitting tightly over the outside of the crucible, and weighs complete about 25 g. Owing to sudden expansion in burning some of the more fluid bitumens, it is well to hold the cover down with the end of the tongs until the most volatile products have burned off.

Some products, particularly those derived from Mexican petroleum, show a tendency to expand suddenly and foam over the sides of the crucible in making this determination, and no method of obviating this trouble without vitiating the result has thus far been forthcoming. Recent experiments in the laboratory of the Office of Public Roads and Rural Engineering indicate that the difficulty may be overcome by placing a small piece of platinum gauze over the sample and about midway of the crucible. The gauze should be so cut or bent as to touch the sides of the crucible at all points, and, of course, is weighed in place in the crucible before and after ignition.

Use of the Determination of Fixed Carbon.—This determination is made on all bituminous products with the exception of tars, upon which reliable results cannot be obtained, owing to the error introduced by the presence of considerable quantities of free carbon.

11. Paraffin Scale. *Apparatus.*—1 one-half pint iron retort (Fig. 61); 1 piece of iron tubing, 30 in. long; 2 100-c.c. Erlenmeyer flasks; 1 500-c.c. (16 oz.) flask, with side-neck for filtering under pressure; 1 freezing apparatus (Fig. 62); 1 6-in. test tube, ¾ in. in diameter; 1 analytical balance, capacity 100 g., sensitive to 0.1 mg.; 1 rough balance, capacity 1 kg., sensitive to 0.1 g.;

1 wash bottle; 1 pint tin cup, seamless; 1 vacuum pump and connections; 1 glass crystallizing dish, 50 mm. in diameter; 1 steam-bath; 1 desiccator with calcium chloride; 1 4-in. steel spatula; 1 bunsen burner with rubber tubing; and 2 iron stands with retort clamp and 1 ring.

Procedure.—Fifty g. of the material under examination should be weighed into the tared iron retort and distilled as rapidly as possible to dry coke. The distillation should be complete in not over 25 minutes. The distillate is caught in a 100-c.c. Erlenmeyer flask, the weight of which has been ascertained previously.

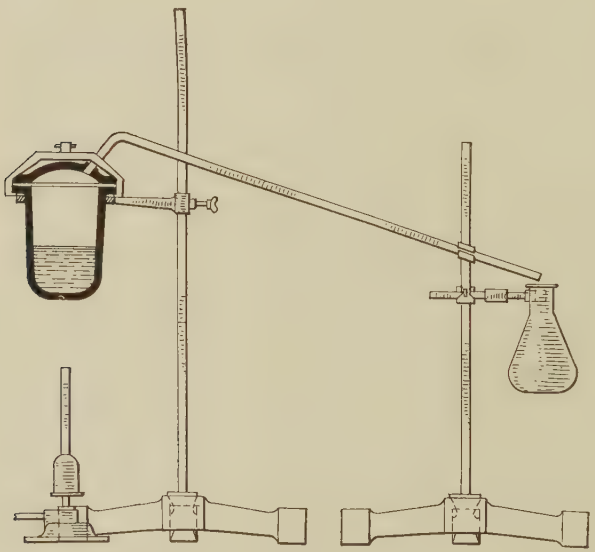

FIG. 61.—Distillation apparatus.

During the early stages of distillation, a cold, damp towel wrapped around the stem of the retort will serve to condense the distillate. After high temperatures have been reached, this towel may be removed. When the distillation is completed, the distillate is allowed to cool to room temperature and then is weighed in the flask. This weight minus that of the flask gives the weight of the total distillate.

The apparatus for freezing out and separating the paraffin scale is shown in Fig. 62. It consists of a bell jar, about 16 cm. high and 14 cm. in diameter, surrounded by a felt or cotton cover. A copper jacket, 4½ cm. in diameter at the top and 21 cm. long,

is held in the neck of the bell jar by means of a rubber stopper. A glass filter-tube fits inside the copper jacket, and, to prevent circulation of air and condensation of water between it and the jacket, a strip of heavy blotting paper is wrapped around the top of the filter-tube. Just below the constriction in the filter-tube a wad of absorbent cotton is placed, tightly compressed to a length of 2 cm. by means of a glass rod. Above this is a wad of tightly packed asbestos wool, about 5 mm. in length, upon which an asbestos filtering mat is prepared. The filter-tube passes through a rubber stopper into a vacuum filtering flask of 500-c.c. capacity. The rubber stopper is placed as tightly as possible against the neck of the bell jar, but, to insure that there is no circulation of air, a disk of blotting paper is compressed between them. The thermometer is capable of recording temperatures from −25° to 0°C. and has the −20°C. graduation at least 14 cm. from the bulb. It is supported by means of a guiding cork and cork disk which is held tightly against the top of the filter-tube by a clamp.

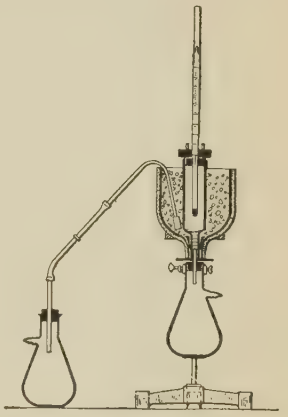

FIG. 62.—Freezing apparatus for determining paraffin scale.

The bell jar is filled with a freezing mixture of ice and salt in the proportion of three to one, which, as it melts, is drawn off through a bent glass tube, which is fitted with a rubber connection and pinchcock, and collected in the 500-c.c. filtering flask. The apparatus is supported on a ring and condenser clamp, attached to a stand.

In separating the paraffin scale, the following procedure is carried out: The filtering flask is removed and a small cork stopper inserted in the lower end of the filter-tube to assist in retaining the solution to be chilled in the upper part of the tube. Ten c.c. of a mixture of equal parts of ethyl ether U. S. P. and absolute ethyl alcohol are poured into the filter-tube, the temperature of which has been reduced to −20°C. From 1 to 2 g. of the well-mixed distillate obtained in the manner previously described then are weighed accurately in a 100-c.c. Erlenmeyer flask, mixed with 10 c.c. of ethyl ether U. S. P., and poured into the filter-tube. Ten c.c. of absolute ethyl alcohol next are placed in the

flask to wash out the ether solution, poured into the filter-tube, and the cover carrying the thermometer placed on the tube. The mixture is maintained at a temperature of −20°C. for 15 minutes, then the cork stopper is removed from the outlet of the filter-tube and the filtering flask is replaced. The corks supporting the thermometer now are loosened and a strong suction is applied to the filter-flask until all the solvent is drawn off. The contents of the filter-tube next are washed with 10 c.c. of a 1 to 1 mixture of ethyl ether U. S. P. and absolute ethyl alcohol, which is chilled to −20°C. in the filter-tube before suction is applied. When the washings have been removed, the vacuum is turned off and the filter-tube removed from the apparatus. The filter-tube then is placed in a clean filter-flask which also contains a 6-in. test-tube in which the dissolved paraffin scale is later collected. About 10 c.c. of warm petroleum ether are poured into the filter-tube and allowed to remain until the paraffin scale has been dissolved. Vacuum then is applied and the dissolved scale drawn into the test-tube. This treatment is followed by two washings, one of 10 c.c. and the other of 5 c.c. of warm petroleum ether, which remove the last traces of paraffin scale. The entire contents of the test-tube then are poured into a weighed platinum or glass crystallizing dish and the petroleum ether is evaporated off over a steam-bath. The dish then is placed in a drying oven maintained at 105°C., until the last traces of petroleum ether have been removed and the paraffin scale has attained a constant weight, after cooling in a desiccator.

The weight of the paraffin scale so obtained, divided by the weight of the distillate taken and multiplied by the percentage of the total distillate obtained from the original sample, equals the percentage of the paraffin scale.

Use of the Paraffin Scale Determination.—The paraffin scale determination may be made on all native bitumens and their products which are suspected of being of a paraffin nature. It is not an extremely accurate determination, however, and is not employed largely.

12. The Extraction of Bituminous Aggregates. *Apparatus for Recovering Aggregate Only.*—1 centrifuge extractor, complete with motor, speed regulator, and electrical connections (Fig. 63); 1 hot-plate; 1 enamel-ware dish, approximately 2 in. deep and 9 in. in diameter; 1 hammer; 1 ¾-in. cold chisel; 1 large metal kitchen spoon; 1 square foot of 1/16 in. deadening-felt

paper; 1½ in. stiff flat brush; 1 500-c.c. bottle or flask; 1 balance, capacity 1 kg., sensitive to 0.1 g.; and 1 sheet of heavy manila paper.

Additional Apparatus for Recovering Bitumen.—1 iron ring-support (ring, 10 cm. in diameter); 1 iron ring-support with condenser clamp; 1 round tin can, 10 by 12 cm., covered with asbestos paper; 1 100-watt incandescent carbon-filament lamp, with socket and connections; 1 asbestos hood (Fig. 64-c); 1 1-liter round-bottom flask, with cork; 1 spiral condenser, length of body 24 cm., with cork to fit, and rubber-tubing connections; 50 cm. of glass tubing, 8 mm. bore; 1 1-liter flat-bottom flask; 1 porcelain evaporating dish, 11 cm. in diameter; 1 watch glass, 20 cm. in diameter; and 1 steam-bath.

Procedure.—The extractor shown in Fig. 63 was designed upon lines suggested by an examination of machines in use by A. E. Schutte and C. N. Forrest. It consists of a ⅕-H.P. 1,100 revolutions per minute vertical-shaft electric motor "*a*," with the shaft projecting into the cylindrical copper box "*b*," the bottom of which is so inclined as to drain to the spout "*c*." A 3/16-in. circular brass plate, 9½ in. in diameter, is shown in "*d*," and upon this rests the sheet-iron bowl "*e*," which is 8½ in. in diameter by 2 5/16 in. high, and has a 2-in. circular hole in the top. Fastened to the inner side of the bowl is the brass cup "*f*," having a circle of ⅛-in. holes for the admission of the solvent, and terminating in the hollow axle, which fits snugly through a hole at the center of the brass plate. The bowl may be drawn firmly against a felt-paper ring "*g*," ¾ in. wide, by means of the 2½ in. milled nut "*h*," for which the hollow axle is threaded for a distance of ¾ in. directly below the upper surface of the plate. The axle fits snugly over the shaft of the motor, to which it is locked by a slot and cross-pin "*i*."

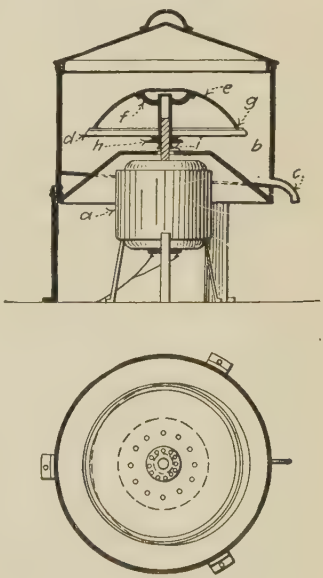

FIG. 63.—Centrifugal extractor (Reeve type).

The aggregate is prepared for analysis by heating it in an enamel-ware pan on the hot-plate until it is sufficiently soft to be thoroughly disintegrated by means of a large spoon. Care must be taken, however, that the individual particles are not crushed. If a section of pavement is under examination, a piece weighing somewhat over 1 kg. may be cut off with hammer and chisel. The disintegrated aggregate then is allowed to cool, after which a sufficient amount is taken to yield on extraction from 50 to 60 g. of bitumen. It is placed in the iron bowl and a ring ¾ in. wide, cut from the felt paper, is fitted on the rim, after which the brass plate is placed in position and drawn down tightly by means of the milled nut. If the bitumen is to be recovered and examined, the felt ring should be treated previously in the empty extractor with a couple of charges of carbon disulphide, in order to remove any small amount of grease or resin that may be present, although a proper grade of felt should be practically free from such products. The bowl now is placed on the motor shaft, and the slot and pin are locked carefully. An empty bottle is placed under the spout and 150 c.c. of carbon disulphide are poured into the bowl through the small holes. The cover is put on the copper box and, after allowing the material to digest for a few minutes, the motor is started, slowly at first in order to permit the aggregate to distribute uniformly. The speed then should be increased sufficiently, by means of the regulator, to cause the dissolved bitumen to flow from the spout in a thin stream. When the first charge has drained, the motor is stopped and a fresh portion of disulphide is added. This operation is repeated from four to six times with 150 c.c. of disulphide. With a little experience the operator soon can gage exactly what treatment is necessary for any given material. When the last addition of solvent has drained off, the bowl is removed and placed with the brass plate uppermost on a sheet of manila paper. The brass plate and felt ring are laid aside carefully on the paper and, when the aggregate is thoroughly dry, it can be brushed on a pan of the rough balance and weighed. The difference between this weight and the original weight taken shows the amount of bitumen extracted. The aggregate then may be tested as occasion requires.

When it is desired to recover and examine the bitumen, the apparatus shown in Fig. 64 will be found convenient and fairly safe for the distillation and recovery of such inflammable solvents

as carbon disulphide. In the laboratory of the Office of Public Roads and Rural Engineering this apparatus is arranged so that the glass tubing passes through a stone partition between two sections of a small hood, thus keeping the distilling and receiving apparatus entirely separated.

The solution of bitumen should be allowed to stand overnight in order to permit the settling of any fine mineral matter that sometimes is carried through the felt ring in the extractor. The solution then is decanted into the flask "*a*," and the solvent is driven off by means of heat from an incandescent lamp until the residue is of a thick sirupy consistency. Meanwhile the solvent is condensed and recovered in the flask "*b*." The residue is poured into an 11-cm. porcelain evaporating dish and evaporated on a steam-bath. The most scrupulous care must be taken at all times that no flames are in its immediate vicinity. Evaporation is carried on at a gentle heat, with continual stirring, until foaming practically ceases. It is advisable to have a large watch-glass at hand to smother the flames quickly should the material ignite. As the foaming subsides, the heat of the steam-bath may be raised gradually, and evaporation is continued until the bubbles beaten or stirred to the surface of the bitumen fail to give a blue flame or odor of sulphur dioxide when ignited by a small gas jet. The dish of bitumen then should be set for about an hour, in a hot-air oven maintained at 105°C., after which it is allowed to cool. Its general character is noted and any tests for bitumens that are necessary are made upon it.

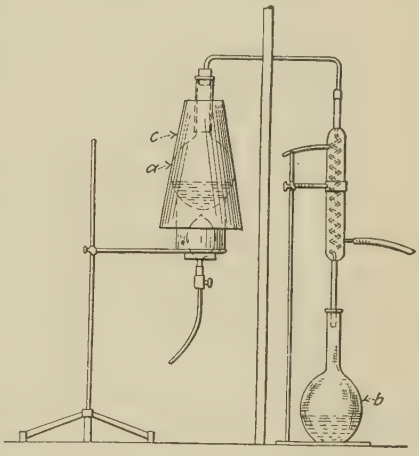

FIG. 64.—Recovery apparatus.

The Recovery of Bitumen.[1]—In recovering the bitumen extracted by carbon disulphide, Richardson recommends the following procedure: Such an amount of crude refined material or old surface is taken as analysis shows will afford about 20 g.

[1] RICHARDSON'S "Modern Asphalt Pavement," 2nd ed.

of pure bitumen. At the same time 20 g. of a bitumen or asphalt cement of corresponding character and of known consistency are taken and treated in the same way as the material under examination. This is done for a control, as will appear. The original material and that of the control determination are placed, in small pieces, in a 600-c.c. Erlenmeyer flask and covered with 300 c.c. of redistilled carbon disulphide. This, after shaking, is allowed to stand overnight or until all lumps are broken down and the bitumen is dissolved. After thorough sedimentation, the solvent is decanted as carefully as possible into a liter flask and 200 c.c. of fresh disulphide are poured on the residue. This should be shaken and allowed to stand again until the insoluble matter has subsided, when the solution of bitumen is decanted as before and added to the first 300 c.c. This process is renewed with several portions of 100 c.c. of carbon disulphide until the residue is clean. The entire solution is allowed to stand overnight, again decanted from the finer sediment of mineral matter, and then swung in a centrifugal machine to remove as much of the still finer mineral as possible. If organic debris is present, the solution must be filtered. In case a more rapid method is desired for old surface mixtures, it is probably quite as satisfactory to swing the solution obtained in the first 300 c.c., as it is, of course, representative of the total bitumen, although only a portion of it.

If no centrifugal is available, the different disulphide solutions are mixed well, allowed to stand for some days, and then decanted. The solutions of bitumen, the one holding that under examination and the control, are, one after the other, placed in the same flask and the solvent distilled off as far as possible at the heat of the steam-bath. The hot and thick residue is poured into an iron dish and placed on a suitable sized hole on the steam-bath and heated. A current of dry steam is blown over the surface of the liquid as long as vapor is evolved. Finally, the presence of the last traces of vapor is tested for with a small flame, such as is used in testing for the flash-point of oils. If all vapor of carbon disulphide which can be distilled in this way has disappeared, the bitumen is in a condition to be brought over a flame or sand-bath and heated, with constant stirring, to a temperature dependent upon its softness, and until it is sufficiently fluid to be poured into a tin box for further treatment.

The bitumen or bitumens under examination and the control bitumen, after having been well identified in the tin boxes, are

brought to the standard temperature and their consistency determined with the penetrometer. The control usually will be found to be softer by twenty or more points than in the original condition. If this is the case, both or all of the extracted bitumens are put in an oven and heated for a length of time to 300°F., depending upon their excess of softness. It is important, of course, that the conditions in the air-bath be uniform.

When the control bitumen has reached its original known consistency, it is assumed that the bitumen or bitumens under examination have done the same thing, and the product is taken as the pure bitumen as it occurs in its original consistency in the crude or refined material or of the cement as it exists in the surface mixture.

13. Grading the Mineral Aggregate. *Apparatus.*—1 set of 8-in. stone-sieves with circular openings of 1½, 1¼, 1, ¾, ½, and ¼ in., respectively; 1 set of 8 in. brass sand-sieves of 10, 20, 30, 40, 50, 80, 100, and 200-mesh, respectively, with pan and cover; 1 rough balance, capacity 1 kg., sensitive to 0.1 g.; 1 1½-in. stiff flat brush; and several sheets of manila paper.

Procedure.—While a mechanical sifter is employed in the Office of Public Roads and Rural Engineering, the following hand method is given for the benefit of those who have not a machine of this character available. When a machine is used, its method of operation should be checked against the hand method described below to obtain practically equivalent results.

For aggregates containing particles too large to pass a 10-mesh screen, the stone-sieves are used, and are stacked in their regular order over a sheet of heavy paper, with the largest size required on top. The weighed amount of stone is placed on the largest sieve and is protected carefully from drafts which might carry away any of the fine material. The upper sieve then is removed from the stack and shaken over a large sheet of paper until no more particles come through. The material thus retained, including any fragments caught in the meshes of the sieve, is weighed and that which passes is added to the contents of the succeeding sieve. This operation is repeated with each succeeding sieve.

When grading sands or fine aggregates, it is customary to take a 100-g. sample in order that the weights may give direct percentages to tenths of 1 per cent. The sieves are stacked in regular order, with the 200-mesh sieve resting on the pan. The sample

is brushed on the top sieve, after which the cover is put on and the stack agitated for about five minutes, with both rocking and circular shaking. Each sieve is removed in order, and is shaken and tapped on a clean piece of paper until no appreciable amount of material comes through. All lumps are broken up by crushing them against the side of the sieve with the finger or a small spatula. The contents of the sieve are emptied into the pan of the balance. All particles caught in the mesh are removed by brushing across the underside of the sieve and are added to the contents of the pan. As great opportunity exists for wide variations in the results of sand gradings made by different persons, owing to the possibility of always getting a little more material to pass by continued shaking, it is well for the novice to repeat his sifting on any given mesh, after having weighed it, in order to see what further loss he can produce. If his judgment has not erred, several minutes' further sifting should not produce a loss of over 0.5 g.

Where coarse aggregates have considerable material passing a 10-mesh sieve and it is desired to grade this material further, it should be weighed and well mixed, quartered, if necessary, and a 100-g. sample should be passed through the sand-sieves. From the percentages so obtained and the weight of material passing the 10-mesh sieve, the percentages of the total aggregate which these finer materials represent may be calculated.

The Office of Public Roads and Rural Engineering has adopted the following recommendations of the Committee on Standard Tests for Road Materials of the American Society for Testing Materials as to the size of wire for standard sand-sieves:

Meshes per linear in.	Diameter of wire, in.	Meshes per linear in.	Diameter of wire, in.
10	0.027	50	0.009
20	0.0165	80	0.00575
30	0.01375	100	0.0045
40	0.01025	200	0.00235

14. Determination of Voids in the Mineral Aggregate. *Apparatus.*—1 1-liter graduated glass cylinder; 1 500-c.c. graduated glass cylinder; 2 pieces of brass tubing (¼ in. bore and 2 in. long); 1 wooden block mounted with soft rubber pad; 1 iron support with condenser clamp; 3 ft. of rubber tubing; 1 pinch-

cock; 1 1½-in. stiff flat brush; 1 small tin scoop; and 2 sheets of manila paper.

Procedure.—The cylinders are prepared for this purpose by drilling a hole in the center of the bottom of each. This hole should be slightly larger than the outside diameter of the brass tubing, one end of which is cemented into it by means of a litharge and glycerin mixture. The upper end of the tube should be flush with the inside of the bottom of the graduated cylinder and

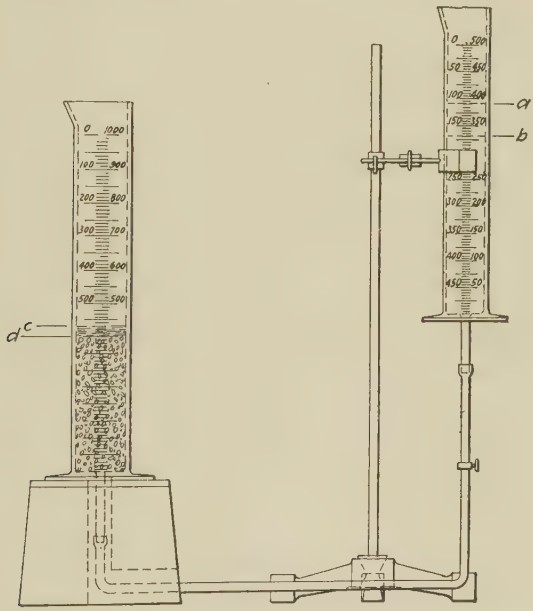

FIG. 65.—Apparatus for the determination of voids in the mineral aggregate

in the large cylinder a piece of 200-mesh wire gauze should be soldered over the end of the tube to prevent fine material passing through.

The apparatus is set up as shown in Fig. 65, with the pinchcock on the rubber tubing closed. The small cylinder is filled with kerosene, after which the pinchcock is opened slowly in order to permit the kerosene to force any air from the tubing and to come flush with the bottom of the large cylinder. The pinchcock then is closed.

The aggregate is mixed thoroughly and quartered, if necessary, until a representative sample of material of at least 300 c.c.

volume is obtained. This sample of aggregate is poured into the large cylinder, a scoopful at a time, with a light tamping of the cylinder against the rubber pad in order to compact the material. The best results usually are obtained by making a cylinder of manila paper inside the glass cylinder, and introducing the aggregate inside the paper. The paper then can be withdrawn slowly while the glass cylinder is being lightly tamped. Segregation of the several sizes of material must be avoided, and any fine material remaining on the paper should be brushed off and added to the aggregate.

When the aggregate in the large cylinder has reached its maximum state of compaction, the volume of kerosene in the small cylinder is read, the pinchcock is opened and the elevation of the small cylinder so regulated as to permit the kerosene to slowly percolate upward through the aggregate, until it has reached a point 20 or 30 c.c. above the top of the aggregate. The meniscus of the kerosene in each cylinder and the volume of the aggregate then are noted. The percentage of voids is calculated as follows:

"a" equals initial volume of kerosene in small cylinder.
"b" equals final volume of kerosene in small cylinder.
"c" equals meniscus of kerosene in large cylinder.
"d" equals apparent volume of aggregate.

Percentage of voids equals $\frac{(a - b) - (c - d)}{d}$

In some cases the kerosene fails to expel all the air from the aggregate, and this fact will be evidenced by bubbles coming to the surface if the aggregate is stirred with a long thin metal rod after the final readings are taken. Accurate results under such conditions are obtained by stirring the aggregate until bubbles cease to appear. This will yield a lower reading on the meniscus of the kerosene in the large cylinder, but the original reading on the volume of rock should be taken.

15. Flash- and Burning-Points.—The flash- and burning-points may be determined by means of the New York State Board of Health tester[1] or by the Cleveland open-cup.[2] The rate of heating is 5°C. per minute.

USE OF THE FLASH- AND BURNING-POINT DETERMINATIONS.—The flash- and burning-point determinations should be made on

[1] See page 74 for a description of this tester.

[2] See page 102.

all bituminous road materials which have to be heated before application and upon all fluid and semi-solid products which show a loss of over 5 per cent. by the volatilization test at 163°C. They also should be made on fluxes which are to be used in cutting hard bitumens.

16. Petrolenes, Asphaltenes and Carbenes.—The components of the bitumen in crude petroleum, petroleum products, malthas, asphalt cements, and solid native bitumens which are soluble in paraffin naphtha, are designated "petrolenes," while those which are insoluble are termed "asphaltenes." That portion of bituminous substances which is insoluble in carbon tetrachloride, but soluble in carbon disulphide, is known as "carbenes.[1]"

The bituminous components of natural and artificial asphalts which are insoluble in paraffin naphtha ("asphaltenes") resemble coal in their behavior to reagents; they contain rather less nitrogen and more hydrogen, but are, unlike coal, soluble in benzene and carbon tetrachloride. The "carbenes" which occur in grahamite are insoluble in carbon tetrachloride and also resemble coal. The resemblance in the deportment of coal and asphaltic substances is ascribed to the common possession of polycyclic structure and similarly linked oxygen.

17. Other Tests Concerning Bituminous Road Materials. Detailed descriptions of other tests, such as ductility, viscosity, distillation, dimethyl sulphate test, flow test, microscopic examination, etc., may be found in such works as Richardson's "The Modern Asphalt Pavement," 1908, Hubbard's "Dust Preventives and Road Binders," 1910, Abraham's "Asphalts and Allied Substances," 1918, and Peckham's "Solid Bitumens," 1909.[2]

18. Specifications for and *other methods* of testing bituminous road materials,[3] and forms for reporting tests, are presented in the *Appendix*, pages 383 *et seq.*

[1] Petroleum asphalts do not show "carbenes" unless they are overheated or over-blown. If more than 0.5 per cent. is present in petroleum asphalts, their quality is to be regarded as questionable.

[2] Other treatises in this field of bitumenology are KÖHLER's "Die Chemie und Technologie der Natürlichen und Künstlichen Asphalte," Braunschweig, 1913; DANBY's "Natural Rock Asphalts and Bitumens," London, 1913; and BOORMAN's "Asphalts, their Sources and Utilizations," New York, 1914.

[3] For analyses of various grades of asphalt, road oil and fluxing oil, see NEWTON and WILLIAMS, *Pet. Age*, **6** (1919), 241.

CHAPTER VI

THE EXAMINATION OF OTHER PRODUCTS OF PETROLEUM[1]

1. Fuel Oils.
2. Gas Oils.
3. Insulating Oils.
4. Cutting Oils.
5. Liquid Petrolatum.
6. Petrolatum.
7. Paraffin Wax.

1. The Examination of Fuel Oils.—The indefinite term *fuel oil* is employed broadly for the description of any product of petroleum which may be used for the production of heat or power, ranging from the distillate series down to and including any product which can be made liquid by steam heat and for which no better market outlet can be obtained. Generally speaking, there are no fixed specifications for fuel oil for industrial purposes, each user being guided by his own peculiar needs and facilities; it must not, however, contain naphtha, and many consumers require that it be of such a consistency that it can be pumped through pipes and burners.[2]

[1] The consideration of such petroleum products as wool oils (cloth oils), leather oils, and floor oils, etc., has been omitted purposely from this Chapter. Those products used by manufacturers of woolens for oiling wool before spinning or rags prior to pulling ("wool oils") consist of fatty oils, oleic acid, and emulsions of oil and oleic acid with small amounts of ammonia or soda in water, also turkey-red oils and soaps; the fatty oils sometimes are replaced in part by mineral oils, and the so-called water-soluble oils are also used (see *Cutting Oils*, page 166). Lewkowitsch, in his "Laboratory Companion" (**1901,** 63), gives analyses of ordinary cloth oils as well as emulsion wool oils. Petroleum leather oils, which, chemically, do not differ from lubricating oils derived from petroleum, have been considered at length by Oberfell in *J. Am. Leather Chem. Assoc.*, **6** (1911), 29; and **11** (1916), 74. On oils used in making leather, see also Bacon, *Drug & Chem. Markets*, **5** (1919), No. 58, 7. On floor oils, especially the use of light lubricating oils for dust-laying purposes, see Heise, *Arbeiten aus dem Kaiserlichen Gesundheitsamt*, **30** (1909), No. 1.

[2] On fuel oils, see Bacon and Hamor, *lib. cit.*, **1,** 117, 119; **2,** 463, 493, 504 and 869.

The examination of fuel oils should include the determination[1] of Baumé gravity, flash-point, fire-point, cold test, and water and B.S.; the percentage of sulphur usually is ascertained, and sometimes the viscosity and heat of combustion are found. The Navy Department specifications for fuel oil (1918) are as follows:

Flash-point by Abel-Pensky or Pensky-Martens closed-cup, not below 150°F.; by Tagliabue open-cup, not below 175°F.

Viscosity, not above 1500 sec. Saybolt at 70°F.

Sulphur, not more than 1.5 per cent.

Water and B.S., not more than 1 per cent.

For railway specifications, see the *Appendix*, page 488.

2. The Examination of Gas Oils.—Gas oil is the fraction intermediate between illuminating oil and lubricating oil.[2] It is adapted especially to the manufacture of oil-gas (which is used to enrich coal-gas for illuminating purposes) and for carbureting water-gas, because of the fact that, being a high-boiling distillate, it will not leave an excessive coke residue on the one hand, while, on the other hand, the molecular weight is sufficiently high to permit ready cracking. Gas oil also is used as a fuel for heavy oil engines. The most important test on the oil is that of distillation, the over-point, distillation-curve to the limit of the thermometer, and residue being secured thereby.[3] Other determinations that may be made are as follows: Baumé gravity, percentage insoluble in petroleum ether (b.p., 40°C.—95°C.), water and sludge, flash- and burning-points, and percentage of sulphur.[4]

"Solar oil" is gas oil from petroleum of the Gulf or Mid-Continent field. An analysis of a typical gas oil is as follows:

Specific gravity, 0.850 at 20°C. (68°F.).

Retort test (bulb of thermometer immersed):

Drop at 260°C. (500°F.).

32 per cent. at 300°C. (572°F.).

60 per cent. at 320°C. (608°F.).

[1] By the methods described in the preceding chapters. See also part 3, of Moore's "Liquid Fuels for Internal Combustion Engines," 1918.

[2] For a select bibliography of gas oils, see *Technical Paper* **120**, Bureau of Mines, 12–14.

[3] See page 12. The distillation should be continued to dryness and the amount of coke determined.

[4] On an "efficiency value" of gas oil, obtained as the product of the heating value and the amount of gas formed, see HEMPEL, *J. Gasbel.*, **53** (1910), 53,77, 101 and 155.

Ignition-point in oxygen, 254°C. (489°F.).
Gross calorific value, 19,584 B.t.u.
Net calorific value, 18,347 B.t.u.
Closed flash-point (Gray), 184°F.
Water, none.
Carbon, 85.9 per cent.
Hydrogen, 12.8 per cent.
Oxygen and nitrogen, 1.09 per cent.
Sulphur, 0.21 per cent.
Ash, none.
Coke, 0.35 per cent.

3. The Examination of Insulating Oils.—Mineral oils are employed as insulating media in several ways. They are used to insulate transformers and switches by immersion, to saturate fibrous and other materials, as in cable work, and in mixing certain insulating paints and varnishes. The desired characteristics of an insulating oil are high resistivity and dielectric strength, low viscosity, high flash-point, chemical neutrality towards metals and insulating materials, freedom from water, sediment

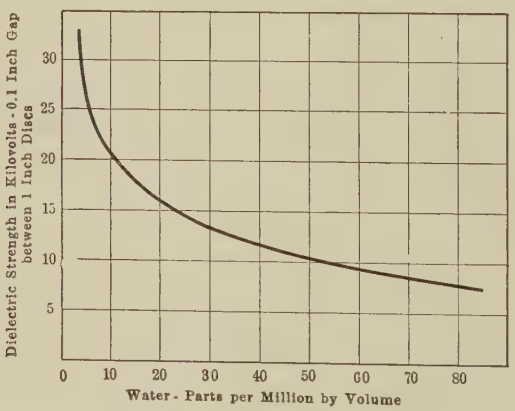

FIG. 66.—The effect of water on the dielectric strength of transformer oil.

and impurities, and chemical stability under local high temperatures.

TRANSFORMER OIL.[1]—An oil suitable for cooling and insulating

[1] On transformer oil, see SKINNER, *Electric J.*, May, **1904**; KINTNER, *ibid.*, October, **1906**; TOBEY, *Trans. Am. Inst. Elec. Eng.*, **29** (1910), 1189; FARMER, *ibid.*, **32** (1913), 2097; *Circular* **36**, U. S. Bureau of Standards; PECK, *Elec. World*, **67** (1916), 1413; HIROBE, OGAWA and KUBO, *ibid.*, **68** (1916), 1301; BACON and HAMOR, *lib. cit.*, **2**, 917; and BATTLE, *Nat. Pet. News*, Dec. 10, **1919**, 41.

transformers should be a petroleum-product unmixed with any other substance. It should be prepared and refined especially for the purpose; should be free from water, suspended matter, acid and alkali, and should contain a minimum of sulphur compounds. According to the Bureau of Standards,[1] the flash-point determined in a closed-cup should not be less than 170°C.; on cold test it should not begin to solidify and no wax should form in the oil above 0°C.; and it should stand a breakdown test of 30,000 volts between spheres of 0.5 cm. radius, 0.40 cm. apart.[2]

The specifications of the Westinghouse Electric and Manufacturing Company are as follows:

"Lectroseal" Transformer Oil

1. This specification covers a specially selected oil which is primarily intended for use in the cases of transformers of the oil-insulated type for the purpose of insulating and cooling the transformer.

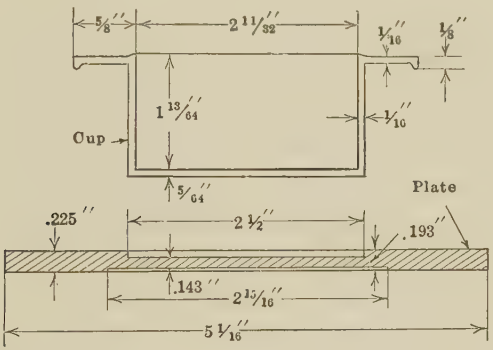

Fig. 67.—Metal plate and test cup used for determining the flash- and fire-points of transformer oils.

I. Physical Properties and Tests

2. The oil must be a pure mineral oil, obtained by fractional distillation of petroleum, unmixed with any other substances. It must not contain moisture, acid, alkali or free sulphur, or any other substance which will affect its electrical insulating qualities.

3. It is desirable that the color of the oil be as light as possible.

4. The flash-point of the oil must not be less than 126°C. (260°F.) and the fire-point must not be less than 149°C. (300°F.). The tests shall be made as follows:

Use metal plate and test cup of the dimensions given in Fig. 67. Fill

[1] *Circular* **22**, 1911.

[2] See also "Standard Handbook for Electrical Engineers," 4th ed., sec. **6**, 55; and Borden, *Elec. World*, **75** (1920), 607.

the cup with oil to within ¼ in. of the rim and remove air bubbles from the surface of the oil by means of a piece of dry paper. The thermometer bulb should be completely immersed, but should not touch the cup.

Adjust the bunsen burner so that the rate of heating of the oil will not be less than 5.5°C. (9.9°F.) nor more than 8°C. (14.4°F.) per minute.

Test for flash-point by passing the gas test flame across the top of the cup, but not allowing it to come in contact with the oil. Apply the test flame about 10°C. (18°F.) below the supposed flash-point of the oil and for every 3°C. (5.4°F.) rise until the flash-point has been reached. This will be indicated when a light bluish flash appears over the surface of the oil upon applying the test flame.

To determine the fire-point, continue heating and testing as described above until the oil takes fire and burns.

5. The oil must have an average breakdown value of not less than 22,000 volts when tested in a device having flat disc electrodes, 1 in. in diameter, with square edges, set with the surfaces in parallel planes 0.1 in. apart, and with their axes in the same horizontal line.

A similar device may be used with discs 0.5 in. in diameter set 0.2 in. apart. The average breakdown value must then not be less than 38,500 volts.

A third device having electrodes consisting of spheres 0.5 in. in diameter set 0.15 in. apart in the same vertical line may be used. The average breakdown voltage with this device must not be less than 40,000 volts.

The following precautions must be followed when using all devices for making dielectric tests, to prevent obtaining results that will cause the condemnation of good oil:

When possible the gap should be adjusted and locked in place first. Then thoroughly rinse with benzine or gasoline. If wiping with cloth is necessary, it must be dry and all such cleaning must be followed by thorough rinsing until all fibers of cotton have been eliminated. Then give a final rinsing with the oil to be tested immediately before placing the test sample in the apparatus.

The temperature of both oil and testing apparatus should be as close together as possible and shall be between 20°C. and 30°C. Lower temperatures are likely to yield low breakdown values and higher temperatures high values.

The oil level for apparatus with disc electrodes shall not be less than one inch above the electrodes and for apparatus with spheres arranged above one another not less than six inches above the center of the gap.

The testing transformer shall have a capacity of at least one K. V. A.

Pour the test sample into the testing device and allow it to stand until all minute air bubbles have had time to escape. This will require two or three minutes.

Apply the testing voltage, starting at a low value and increasing steadily at the rate of about 3000 volts per second without opening the circuit until breakdown takes place.

After each break, jar the testing device but do not cause the oil to be agitated sufficiently to take up air bubbles. This will tend to loosen particles of carbonized oil from between the electrodes. Do not introduce any

device into the oil for stirring, as it is likely to cause low breakdown voltages on account of introducing minute particles of foreign matter or moisture. Jarring will not remove all carbon from between the electrodes, but the remainder will be driven off by the next application of voltage before the breakdown value is reached.

Repeat the application of voltage until five breakdowns have been made and use the average value as the breakdown voltage of the oil.

Sometimes a small bright spark passes across the gap but does not form an arc or cause circuit breakers or fuses to open the circuit. Such discharges should be disregarded in recording results. They are not likely to occur if sufficient time is allowed for all air to escape before applying voltage. It is better to make tests on several samples of oil if the first sample has a low breakdown value. If the tests on any one sample from the same tank, or other container, yield satisfactory results and the tests on other samples are low, the latter may be disregarded as having been caused by contamination in taking from the container or by insufficient cleaning of the testing device.

6. The viscosity shall not be more than 40 seconds at 40°C. (104°F.), as determined by means of a Saybolt viscosimeter.

7. The oil must not show a deposit or any change other than a darkening of color, after being tested in the following manner: Place two ounces of oil in a 6-ounce glass beaker and heat with bunsen burner gradually and uniformly at the rate of 3.5°C. (6.3°F.) per minute until a temperature of 232°C. (450°F.) is reached, then allow to cool. Put the oil in a tall bottle or large test tube and allow to stand for 12 hours at room temperature, then hold toward a strong light and examine for deposit.

8. A sample shall be taken from one drum for each 25 drums received, and from each tank car received, the oil being taken from the lowest part of the drum or tank car. Great care must be taken to keep the sample of oil free from moisture and dirt. The sneaks and bottles used for sampling should be carefully cleaned with benzine and dried before using, and bottles kept corked before and after sample has been obtained.

II. Packing and Marking

9. Shipment of oil must be made in tin cans, iron barrels, or tank cars, as specified on the individual order. Wooden barrels may be called for in special cases. All cans used for shipment of oil must be new, clean and dry; all barrels must be clean and dry, and be reasonably free from rust before filling. All tin cans must be hermetically sealed by soldering, and must be properly boxed for shipment. The bungs and taps of iron drums must be made oil-tight by means of lead washers, bung being screwed tight so that the washer is under compression.

10. All iron barrels must be sealed by means of a substantial car seal of standard form, the wire of seal being so arranged that the bung or cap cannot be opened without breaking the seal.

11. (*a*) Each drum shall be plainly marked as follows:

Requisition number.
P. D. Spec. 1651-D.
No. of gallons.
Name of manufacturer.

For example.—Req. 0-5100; P. D. Spec. 1651-D; 50 gal.; Blank Oil Company.

(*b*) Each drum shall bear a serial number stamped into the metal of the drum or on a metal plate soldered to the end of the drum.

12. Each tank car shall have a packing slip with the following information:

Requisition number.
P. D. Spec. 1651-D.
No. of gallons.
Name of manufacturer.

For example.—Req. 0–5000; P. D. Spec. 1651–D; 6000 gal.; Blank Oil Company.

III. Inspection

13. Unless otherwise specified, inspection shall be made at destination. Inspection may be made at point of manufacture, if desired by the purchaser.

IV. Rejection

14. The Westinghouse Electric and Manufacturing Company reserves the right to reject any portion or all of the material which does not conform to the above specification in every particular, and to return the rejected material to the manufacturer or seller for full credit at price charged, f.o.b. point of delivery specified by the purchaser. If the material is to be replaced, a new order will be entered at prices, terms and conditions acceptable to the purchaser.

The General Electric Company specifies that:

(1) "Transil Oil No. 8" must be pure mineral oil, of which the characteristics shall be as follows:

Specific gravity at 15.5°C., approximately 0.83.
Cold test, not higher than −2°C.
Flash-point, not less than 130°C.
Fire-test, not less than 149°C.
Viscosity, not higher than 39 sec. at 40°C.
Reaction—neutral.
Ash and saponification value—*nil.*

The viscosity shall be determined in the Saybolt cup at a temperature of 40°C. The flash-point and fire-test shall be determined in the Vacuum Oil Company's standard open-cup flash-tester. The cold-test shall be determined by chilling 30 c.c. of oil in a 4-oz. vial, 1 in. in diameter, with a thermometer immersed to the center of the oil. The chilling of the oil may be done with any suitable freezing medium. The vial containing the oil must not be in direct contact with the freezing medium, but shall be surrounded by an air space, which shall be large enough so that on further cooling, after the oil has come to 5°C., the temperature shall not drop faster than 1°C. per minute. Every 3°C. below this point, tip the jar to an angle of about 15° from the vertical. The last point at which the oil will flow when the tube is held in this position and its base gently tapped on the palm of the hand is taken as the cold test.

(2) The oil is to be used as an electrical insulator, and it is, therefore, essential that it be kept as nearly as possible free from moisture, acid, sulphur and from all other foreign substances which might in any wise affect or endanger its electrical insulating qualities or react with metals used in the construction of electrical apparatus. Tests to determine the electrical insulation of the oil will be made by the General Electric Company in one of their standard testing outfits which are shown on Drawing M-545089, with a gap between terminals of 0.200 in. No. 8 oil must have a disruptive strength of not less than 40,000 volts. Test may also be made in one of

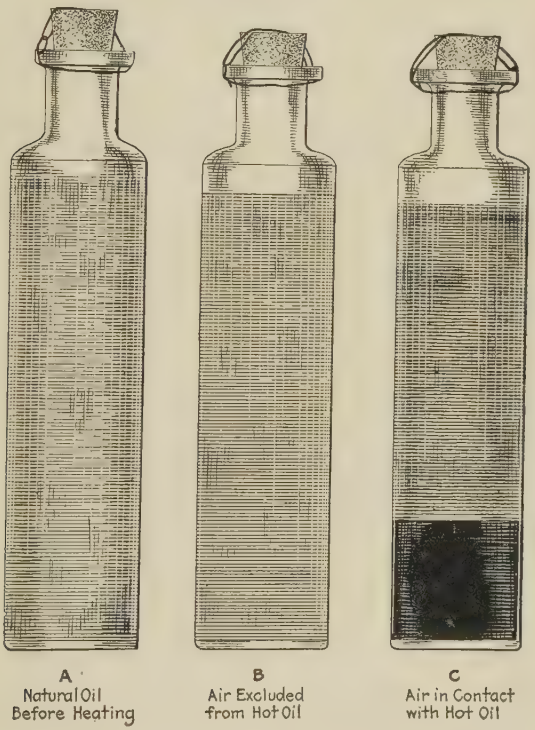

FIG. 68.—The effect of air on hot transformer oil. Test at 130°C. for 18 days.

the standard testing outfits shown on Drawing P-568839, with a gap between terminals of 0.100 in. No. 8 oil must have a disruptive strength of not less than 23,000 volts when tested in this manner.

(3) At any time within one year from the date of receipt the oil must show no precipitate when heated to 120°C. for 72 hours and pure oxygen allowed to bubble through.

The solid sludge formed in the high-resistance insulating mineral oils used in transformers is undoubtedly an oxidation product resulting from the direct action of the air, often catalytically

accentuated by the presence of metals and especially of copper. Possibly also ozone, formed by electric discharges from the transformers, plays a part, while oils containing large proportions of olefines and other unsaturated hydrocarbons are presumably more liable to sludge-formation than oils which are free from these substances.[1] Recent experiments upon the question of sludge formation appear to show that the oxidation of petroleum oil, as used in highly insulated transformers, by air at temperatures up to 100°C., is not of the nature of a substitution, but is chiefly an additive reaction, at least in so far as the hydrogen is concerned, for no water is formed, or, if formed, it is only in very small quantities, very much less than is produced, for instance, when coal is oxidized under similar conditions.

4. The Examination of Cutting Oils.—Compounded oils, or cutting oils, are manufactured in great variety for the metal-cutting and stamping industries and for drilling, boring, reaming, milling and various other machine-shop operations. They may be subdivided roughly into two general classes, viz., *straight* cutting oils and *soluble* cutting oils. The straight cutting oils, which consist of lard oil, sperm oil, or vegetable oils, or mixtures of one or more of these oils with certain mineral oils,[2] are used directly on the machines without any further treatment and are intended principally for the more delicate lathe work and fine boring and reaming operations and also for work on high-carbon, special alloy steels and other hard metals. Lard oil, or a mixture of lard oil and mineral oil, is the most extensively used cutting oil of this class.

Soluble cutting oils are of many types, but all depend upon the property of forming an emulsion when mixed with water. These products are used primarily for heat conduction in heavy cutting, drilling, milling, stamping and other operations and at the same time they furnish considerable lubrication at the cutting points and surfaces.[3] They are compounded usually from mineral oils mixed with a specially treated animal or vegetable oil, such as lard, cottonseed, corn, rapeseed, castor, olive, rosin, neatsfoot,

[1] Hence the iodine value is held to be of importance with compressor lubrication (Moore, *The Engineer*, Aug. 20, **1915**) and transformer oil (Digby, *J. Inst. Elec. Eng.*, **53**, No. 239, 146), where it is an indication of the liability to oxidation. On the determination of the iodine values of mineral oils, see pages 20 and 59.

[2] Pure mineral oil is used only to a limited extent as a straight cutting oil.

[3] See Hammond, *Machinery*, January, **1917.**

and other oils. They are known as sulphonated, phenolated and soap oils.

Copeland[1] has discussed at length the methods for the analysis of compounded oils. He describes procedures for the separation of the saponifiable matter and the mineral oil and for conducting physical tests (emulsification, segregation, and evaporation) on cutting oils of both classes.

METHODS OF ANALYSIS

Two general methods, designated as A and B, were devised by Copeland for the analysis of compounded oils. Method A is a gravimetric separation of the saponifiable matter and the mineral oil and is useful as a check method and also where a further examination of the separated oils is contemplated, to determine their origin, nature and characteristics. Method B is a volumetric method and gives more complete information as to the condition of the saponifiable matter; it is also much more rapid. Both methods can be applied to the two classes of oils without any changes.

In the following methods, all flasks and other containers used for weighing samples and residues first should be thoroughly cleaned, rinsed with distilled water, alcohol, and ether, and then dried in an oven for one hour at a temperature of 100° to 105°C.

Preparation of Sample.—Mix the oil thoroughly by vigorously shaking the container, or, if in a barrel, by rolling it on the bilge; then fill 4-ounce oil sample bottles with the well-mixed oil and label properly.

Before commencing the analysis, the color, odor, turbidity, and any other characteristics of the original oil should be noted.

Method A.—*Determination of Saponifiable Oil and Mineral Oil.*

Weigh 4 to 6 g. of the thoroughly mixed oil in a 300-c.c. Erlenmeyer flask, add 50 c.c. of alcoholic potash (40 g. of potassium hydroxide dissolved in one liter of alcohol that has been redistilled after standing for some time over potassium hydroxide), and then boil the mixture under a reflux condenser for from one to two hours. An oil- or water-bath maintained at about 100°C., or an electric hot-plate, is suitable for boiling the liquid.

Disconnect the apparatus and boil off most of the alcohol, being careful not to overheat the oil or decomposition may result.

[1] *Met. Chem. Eng.*, **17** (1917), 27.

Add about 50 c.c. of hot water to the residue in the flask, agitate and transfer to a 500-c.c. separatory funnel, and wash out the contents of the flask with small successive portions of hot water, keeping the total volume in the funnel under 150 c.c. Cool the solution in running water and when cold add 75 c.c. of ether, stopper the funnel and shake vigorously. Allow the two layers to separate for about 12 hours or over night. If, after standing for some time, the solution does not show a tendency to separate into two distinct layers, the separation may be accelerated by adding a little alcohol, about 2 c.c. at a time, and shaking after each addition, which usually will effect a complete separation. The upper layer contains the ethereal solution of the unaffected mineral oil and the lower layer contains the animal or vegetable oil which has been converted into water-soluble soap by saponification with the alcoholic potash.

Run the lower layer into another separatory funnel and wash the ethereal solution with about 50 c.c. of cold water, allowing the water to run down the sides of the funnel while rotating the same; allow to clear and then run the wash water into the second funnel. Add dilute sulphuric acid, containing about 5 c.c. of concentrated acid, to the soap solution in the second funnel, to liberate the fatty acids. Cool in running water and when cold add 75 c.c. of ether, stopper the funnel and shake vigorously, allow to separate for about one-half hour or until clear, and then run off the lower aqueous layer, which is discarded.

Wash the ethereal solution of the fatty acids with about 50 c.c. of cold water, allowing the water to run down the sides of the funnel while rotating the same; allow to clear, run off, and repeat the washing with cold water until the wash water is no longer acid to blue litmus paper; about two washings will suffice.

Saponifiable Oil.—Drain off the wash water entirely from the ether solution of the fatty acids, and then run the ethereal solution into a weighed 150-c.c. Erlenmeyer flask and rinse out the funnel with a small amount of ether. Cautiously evaporate off the ether and then heat the flask for one hour in a drying oven at a temperature of 100° to 105°C., cool and weigh as saponifiable matter. The saponifiable matter in this case consists of the hydrates of the fatty acids. If any water is carried over into the ether solution, it may be removed when heating the residue, by spreading the oil in a thin layer over the interior of the flask; any

water present then is volatilized more easily without causing the oil to spurt.

Mineral Oil.—Drain off the wash water entirely from the ethereal solution of the mineral oil in the first funnel, and then run the ethereal solution into a weighed 150-c.c. Erlenmeyer flask and rinse out the funnel with a small amount of ether. Proceed with the evaporation, heating, etc., exactly as directed for the determination of the saponifiable oil, finally weighing the product as mineral oil.

METHOD B.—*Determination of Total Saponifiable Matter and Total Fatty Acids.*

Weigh 4 to 6 g. of the oil in a 300-c.c. Erlenmeyer flask, add 50 c.c. of the alcoholic potash solution and boil the mixture under a reflux condenser for from one to two hours. Disconnect the apparatus, boil off most of the alcohol, add about 50 c.c. of hot water to the residue, agitate and transfer to a 500-c.c. separatory funnel and wash out the flask with small successive portions of hot water. Add dilute sulphuric acid, containing about 5 c.c. of concentrated acid, to the solution in the funnel and cool in running water; when cold, add 75 c.c. of ether, stopper and shake vigorously. Allow the two layers to separate for about one-half hour or longer if necessary; when clear, run off the lower aqueous layer and discard. Wash the ethereal solution with about 50 c.c. of cold water, allowing the water to run down the sides of the funnel while rotating the same, being careful not to disturb the ethereal layer too much, as troublesome emulsions are liable to be formed. Allow to stand until clear, run off the wash water, discard and repeat the washing with cold water until the wash water is free from acid when tested with blue litmus paper; about two washings will suffice. Drain the ethereal solution into the original flask, which has been rinsed with a little alcohol and allowed to drain, and wash out the funnel with a small amount of ether. Cautiously evaporate the solution in the flask until most of the ether has been driven off.

Neutralize 50 c.c. of alcohol with tenth-normal alkali, using 20 drops of a 1 per cent. solution of phenolphthalein as an indicator. Add the neutral alcohol to the contents of the flask, heat to boiling and titrate with the tenth-normal alkali until the pink color persists after vigorous shaking. The flask should be stoppered and shaken vigorously toward the end of the titration.

Total Saponifiable Matter.—Multiply the number of cubic

centimeters of tenth-normal alkali used in this titration by 0.028-773 and divide by the weight of the sample taken, to obtain the total saponifiable matter in one gram of the oil.

In this titration it is assumed that one gram of the saponifiable matter requires 195 milligrams of potassium hydroxide for its saturation, *i.e.*, it has a saponification value of 195. One cubic centimeter of tenth-normal alkali is equivalent to 0.0056108 gram of potassium hydroxide, consequently—

$$\frac{0.0056108}{0.195} = 0.028773.$$

This factor is accurate for most of the oils entering into this class of compounds; exceptions will be noted later.

Total Fatty Acids.—Multiply the number of cubic centimeters of tenth-normal alkali used in this titration by 0.0282 and divide by the weight of the sample taken, to obtain the total fatty acids in one gram to the oil.

The above factor is based on the assumption that the fatty acids exist as oleic or some similar acid, which is a very close approximation of the truth; exceptions will be noted later.

Determination of Free Fatty Acids.

Weigh 5 to 10 g. of the oil in a 300-c.c. Erlenmeyer flask Neutralize 50 c.c. of alcohol with tenth-normal alkali, using 20 drops of phenolphthalein as an indicator. Add the neutral alcohol to the contents of the flask, heat to boiling and titrate with the tenth-normal alkali until the pink color persists after vigorous shaking. The flask should be stoppered and vigorously shaken toward the end of the titration.

Free Fatty Acids.—Multiply the number of cubic centimeters of tenth-normal alkali used in this titration by 0.0282 and divide by the weight of the sample taken, to obtain the free fatty acids, as oleic acid, in one gram of the oil.

Determination of Soap, Fatty Acids Combined as Soap, Fatty Acids Combined as Fat, and Neutral Oil.

Weigh 4 to 6 g. of the oil in a 300-c.c. Erlenmeyer flask, add about 50 c.c. of hot water, agitate, transfer to a 500-c.c. separatory funnel and wash out the flask with small successive portions of hot water. Add dilute sulphuric acid, containing about 5 c.c. of concentrated acid, to the solution in the funnel and cool in

running water. When cold, add 75 c.c. of ether, stopper the funnel and shake vigorously. Allow the two layers to separate for about one-half hour or longer if necessary; when clear, run off the lower aqueous layer and discard. Wash the ethereal layer with about 50 c.c. of cold water, allowing the water to run down the sides of the funnel while rotating the same. Allow to stand until clear, run off the wash water and repeat the washing with cold water until the wash water is no longer acid when tested with blue litmus paper; about two washings will suffice. Run the ethereal solution into the original flask, which has been previously rinsed with a little alcohol and allowed to drain, and carefully evaporate off most of the ether.

Neutralize 50 c.c. of alcohol with tenth-normal alkali, using 20 drops of phenolphthalein as an indicator. Add the neutral alcohol to the contents of the flask, heat to boiling and titrate with the tenth-normal alkali until the pink color persists after vigorous shaking of the stoppered flask.

Fatty Acids as Soap.—Multiply the number of cubic centimeters of tenth-normal alkali used in this titration by 0.0282 and divide by the weight of the sample taken and call the result (*a*). Subtract from this result the free fatty acids, per gram of oil, as found in a previous determination, and the remainder is the fatty acids combined as soap in 1 gram of the oil.

Soap.—Multiply the result obtained for the fatty acids combined as soap by 1.078; the product is the sodium soap formed by the alkali and these acids.

Sodium soap (sodium oleate), mol. weight.

$$\frac{304}{282} = 1.078.$$

Fatty acids (oleic acid), mol. weight.

$$\frac{304}{282} = 1.078.$$

If a potassium soap is required, substitute the factor 1.13 in the above calculation.

Fatty Acids as Fat.—Subtract the result (*a*) from the total fatty acids found in a previous determination, to obtain the fatty acids combined as fat in 1 gram of the oil.

Neutral Fatty Oil.—Subtract the result (*a*) from the total saponifiable matter found in a previous determination, to obtain the neutral fatty oil in 1 gram of the sample.

NOTES.—All the determinations in the foregoing methods of analysis are applicable and useful in the examination of soluble cutting oils, but as straight cutting oils obviously do not contain soap nor fatty acids as soap, these determinations are omitted. There are actually only three determinations in Method B, the other information being obtained by simple calculations. Much time can be saved if all the titrations are performed at one time and several oils can be analyzed successively, leaving the titrations to the last. The end point in these titrations is usually very sharp; but if any trouble is experienced in accurately determining this point with dark-colored oils, by titrating back with tenth-normal hydrochloric acid the change can be rendered very distinct, so that one or two drops of either standard solution will give the color reaction. It is advisable to follow this procedure in all the titrations, as very concordant results can be obtained and over-titrations avoided.

As Method A gravimetrically determines only the hydrates of the fatty acids that exist in the oil, the results by this method will be a trifle low for oils where these fatty acids were originally present as neutral fatty oil, due to the loss of the glycerol radicle. This loss is somewhat compensated for, due to the fact that the fatty acids are weighed as hydrates and not as anhydrides.

In Method B the total saponifiable matter is based on the assumption that the oil has a saponification value of 195, which is correct for most of the oils entering into this class of compounds. Sulphonated oils, however, usually contain a castor oil base, which has a saponification value of about 184; consequently—

$$\frac{0.0056108}{0.184} = 0.0305.$$

This factor would be the proper one to use when castor oil is required and 0.02983 would be the correct factor for the ricinoleic acid of this oil.

5. The Examination of Liquid Petrolatum.—Two grades of liquid petrolatum (*petrolatum liquidum;* liquid paraffin; medicinal oil; pharmaceutical oil; or "mineral oil") are recognized by the ninth revision of The *Pharmacopœia of the United States of America,* namely:

HEAVY LIQUID PETROLATUM.—Heavy liquid petrolatum has a

viscosity of not less than 3.1 when determined by the test given below.

Light Liquid Petrolatum.—Light liquid petrolatum has a viscosity of not more than 3 when determined by the test given below and vaporizes freely.

Each variety conforms to the following description and tests:[1]

Specific gravity for liquid petrolatum, 0.828 to 0.905 at 25°C. A colorless, transparent, oily liquid, free, or nearly free, from fluorescence, odorless and tasteless when cold, and possessing not more than a faint petroleum odor when heated. When cooled to 10°C., liquid petrolatum does not become more than opalescent (solid paraffins). Insoluble in water or ethyl alcohol; soluble in ethyl ether, chloroform, benzine, or in fixed or volatile oils. Camphor, menthol, thymol, and many similar substances are dissolved by liquid petrolatum. Boil 10 mils[2] of liquid petrolatum with an equal volume of alcohol; the alcoholic liquid is not acid to litmus (acids). Introduce into a glass-stoppered cylinder, which has been rinsed previously with sulphuric acid, 5 mils of liquid petrolatum and 5 mils of colorless sulphuric acid, heat in a water-bath during ten minutes, shaking well at intervals of thirty seconds; the oil remains unchanged in color and the acid does not become darker than pale amber (carbonizable impurities). Prepare a clear, colorless saturated solution of lead oxide in an aqueous solution of sodium hydroxide (1 in 5), and mix 2 drops of this solution with 4 mils of liquid petrolatum and 2 mils of dehydrated ethyl alcohol; the mixture does not darken after heating for ten minutes at 70°C. and cooling (sulphur compounds).

Viscosity.—Make a permanent mark about 2 cm. below the bulb of a 50 mil pipette of the usual type and note the time, in seconds, required at 25°C. for the level of distilled water to fall from the upper to the lower mark as the liquid flows from the pipette. The time should not be less than twenty-five seconds nor more than thirty seconds for the pipette selected. Draw the liquid petrolatum to be tested into this pipette, which should be

[1] For a general account of the preparation and properties of liquid petrolatum and its uses in medicine, see Bacon and Hamor, *lib. cit.*, **2**, 634 and 898.

[2] A "mil," or milliliter, is the thousandth part of a liter. It is but slightly different from a cubic centimeter, for one liter is equivalent to 1.000027 cubic decimeters.

clean and dry, and note the time, in seconds, required at 25°C. for its level to fall from the same upper to the lower mark as used for the water. Divide the number of seconds thus noted by the number of seconds required for water to fall from the upper to the lower mark, as above determined. The quotient indicates the viscosity. Distilled water at 25°C. is taken as 1.

6. The Examination of Petrolatum.—Petrolatum (petroleum jelly or soft petroleum ointment) is marketed in three grades: amber (crude, technical or veterinary petrolatum), light yellow, and white (*petrolatum album* or white petroleum jelly) or decolorized.[1] It is used widely in medicine as an emollient and ointment-base, and accordingly the pharmacopœial standards are presented herewith.

PETROLATUM.[2]—A purified mixture of semi-solid hydrocarbons, obtained from petroleum. Petrolatum is an unctuous mass, varying in color from yellowish to light amber, having not more than a slight fluorescence, even after being melted; it is transparent in thin layers; completely amorphous; free or nearly free from odor or taste. Petrolatum is insoluble in water; almost insoluble in cold or hot ethyl alcohol, or in cold dehydrated ethyl alcohol; freely soluble in ethyl ether, chloroform, carbon disulphide, turpentine, benzine, benzene, or in most fixed or volatile oils. Specific gravity, 0.820 to 0.865 at 60°C. It melts between 38° and 54°C.

Heat about 2 g. of petrolatum in an open porcelain or platinum dish, over a bunsen flame; it volatilizes without emitting an acid odor, and on incineration not more than 0.05 per cent. of ash remains. Shake melted petrolatum with an equal volume of hot distilled water; the latter remains neutral to litmus (acids or alkalies). Digest 10 g. of sodium hydroxide and 50 mils of distilled water, then separate the aqueous layer and supersaturate it with sulphuric acid; no oily or solid substance separates (fixed oils, fats, or rosin).

PETROLATUM ALBUM.—White petrolatum is a white or faintly yellowish unctuous mass, transparent in thin layers, even after cooling to 0°C.; it is completely amorphous. In other respects white petrolatum has the characteristics of and responds to the tests for identity and purity under petrolatum (*supra*).

[1] On the chemistry of petrolatum, see BACON and HAMOR, *lib. cit.*, **2**, 631–643.

[2] "The Pharmacopœia of the United States of America," 9th rev., 313.

7. The Examination of Paraffin Wax.—Paraffin,[1] in its refined condition, is a white,[2] waxy, inodorous, tasteless substance, harder than tallow, softer than vegetable wax, with a specific gravity of 0.890. Its melting-point is variable, depending somewhat upon its origin; it ranges between 43° and 65°C. (109° and

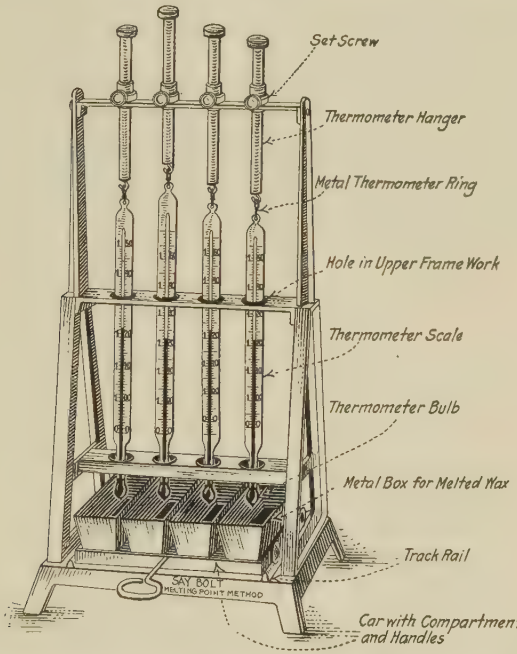

FIG. 69.—The Saybolt improved apparatus for the determination of the melting point of paraffin wax.

The melted wax is poured into the metal box, and when the latter is three-fourths filled the thermometer is adjusted so that the bulb is well covered by the wax. The car then is moved back and forth until a point is reached when the mercury column remains stationary for about one minute. This temperature represents the melting-point of the wax.

151°F.), but usually the refined paraffin wax of commerce is of two melting points, *viz.*, 118°–120°F. and 126°–128°F.

[1] On the technology of paraffin, see BACON and HAMOR, *lib. cit.*, **2**, 478, 483, 753 and 894.

[2] In the course of its manufacture paraffin wax generally is examined in the liquid condition for color, and this may be done conveniently by heating the cell of a Lovibond tintometer, filling it with hot wax, and examining it before it begins to solidify. Liquid wax showing 0.74 yellow (or less) in an 18-in. stratum will be of satisfactory color when solid. On the determination of the *structure* of paraffin wax, see MAPES, *Science*, **52** (1920), 256.

THE DETERMINATION OF MELTING-POINT.—For the determination of the melting-point of wax the following method generally is employed in the British oil trade: A test-tube about 1 in. in diameter is filled to the depth of about 2 in. with the melted paraffin, a small thermometer is inserted, and the whole steadily stirred, while the test-tube and its contents are allowed to cool slowly. The temperature at which the thermometer remains stationary for a short time is the melting (setting) point. Fig. 69 illustrates apparatus for the application of a method which is an extension of the British method.

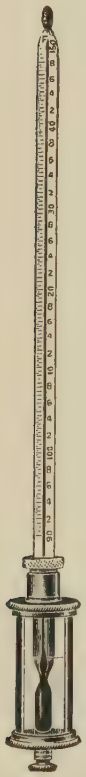

FIG. 70.—The "Tag" melting-point tester for paraffin wax.

The *American method* is carried out as follows:

Apparatus.—Hemispherical dish, 3¾ in. in diameter; bunsen burner; and wax-testing thermometer.

Procedure.—The dish is filled ¾ full of the melted paraffin. The thermometer is suspended in the center of the dish with the bulb ¾ immersed. The melted paraffin is allowed to cool until a film extends from the sides of the vessel to the thermometer. The temperature then is read and recorded as the melting-point.[1]

THE DETERMINATION OF OIL AND WATER.[2] *Apparatus.*—Moore standard wax-testing press (see Fig. 71); four circular botters, 5½ in. in diameter; two circular pieces of fine linen cloth, 5½ in. in diameter; and an analytical balance.

Procedure.—Pulverize some of the wax and weigh out about 32 g. Place two blotters and then one piece of linen in the bottom of the cup and carefully spread the paraffin on the latter. Cover the paraffin with the other piece of linen, followed by the remaining blotters. Finally insert the plunger and apply the maximum pressure for 5 minutes. For accurate results, the paraffin should be kept at a temperature of 60°F. The percentage of oil and water is found by weighing the wax after the test or by ascertaining the increase in weight of the blotters.

[1] In some laboratories it is the practice to determine the melting-point by the British method and to add 3°F. to that value.

[2] For an account of the development of wax-testing presses, see BACON and HAMOR, *lib. cit.*, **2**, 766. The same authors describe an experimental wax-press, *ibid.*, 758.

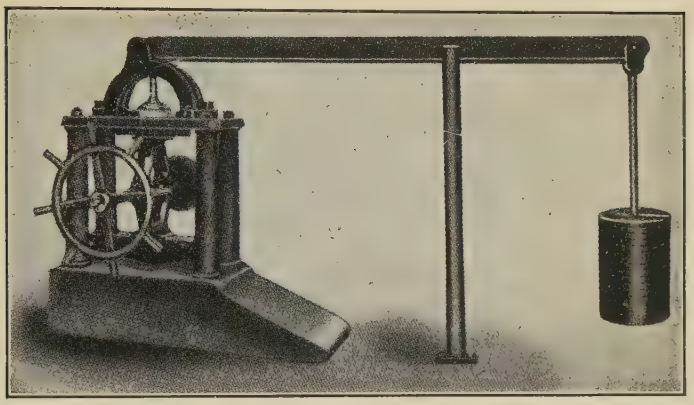

FIG. 71.—Moore's standard wax testing press.

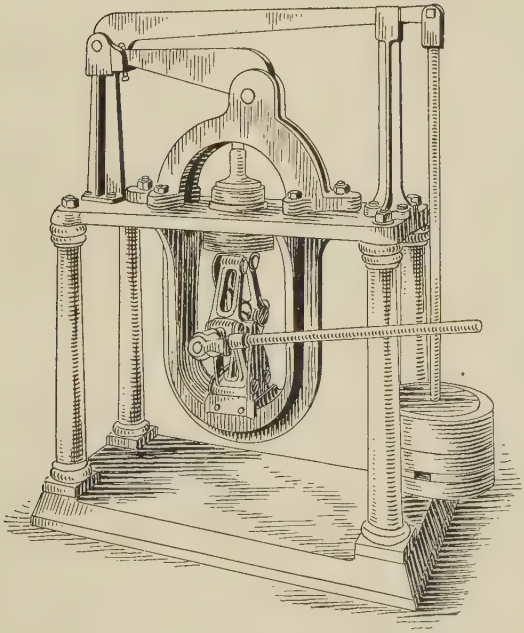

FIG. 72.—The wax testing machine manufactured by the Carbondale Instrument Company, Carbondale, Pa.

Other "Mineral Waxes."—It may be noted in passing that paraffin from petroleum may be distinguished from paraffin from shale-oil, brown-coal tar, etc., by a method devised by Marcusson and Meyerheim.[1] Ozocerite (ozokerite, "native paraffin," or "mineral tallow"),[2] which occurs in a commercially important deposit in Wasatch County, Utah,[3] is tested for ceresin-yield according to the procedure of Lach and von Boyen.[4] Montan wax, which is found in German brown coal in amounts varying from 1 to 27 per cent., has been described at length by Meyerheim.[5]

[1] *Z. angew. Chem.*, **23** (1910), 1057.

[2] Christmatite, hatchettite, helenite, urpethite, and zietrisikite are hydrocarbon minerals near ozocerite.

[3] *Met. Chem. Eng.*, **16** (1917), 246. For a bibliography of ozocerite, see Gosling, *School of Mines Quart.*, **16** (1894), 41.

[4] *Z. angew. Chem.*, **11** (1898), 383; and Holde-Mueller's "Examination of Hydrocarbon Oils," **1915,** 243. On the examination of ceresin, see Ulzer and Sommer, *Chem.-Ztg.*, **30** (1906), 142; and Marcusson and Schlüter, *ibid.*, **31** (1907), 348.

Seifenfabr., **39** (1919), 365 and 394.

CHAPTER VII

THE SAMPLING OF PETROLEUM OILS[1]

The sampling of oil or of any other commodity for the purpose of testing or analysis is a matter of the utmost importance. In fact, it is just as essential to obtain a sample which represents the bulk of the product from which it is taken, as it is to make an accurate analysis of the product; and probably as much confusion and controversy have been occasioned by samples which are not representative as by the use of improper technic and unreliable methods in testing.

In taking samples of oil, which are to represent an average of that contained in a tank or other receptacle,[2] it must be remembered that, at times, a stratification of oils of different gravities and other properties may obtain. This is true in cases where several oils have been pumped into a tank for blending purposes, and have not been mixed properly by agitation, especially if the oils are of different densities or are very viscous. Oil of heavier gravity will, of course, lie nearer the bottom than oil of lighter gravity; but other tests, such as boiling-point, cold test, viscosity, etc., may vary at different levels. If convenient, it is always advisable to agitate the oil with air or other means before sampling.

SAMPLING FROM TANKS

The most practical method for taking samples from tanks is to obtain them directly in the bottles from which they are to be tested. This may be done by using a weight heavy enough to sink a pint or a quart bottle attached to a cord sufficiently long to reach to the bottom of the tank. The cord is so attached to the bottle to keep it in an upright position; this can be done by half-hitches.

[1] Contributed by W. E. Espy, Cosden and Company, Tulsa, Okla.

[2] The methods of sampling fatty oils, recommended by the committee on the analysis of commercial fats and oils of the American Chemical Society, are of suggestive aid to the petroleum chemist (see *J. Ind. Eng. Chem.*, **11** (1919), 1161).

To take an average sample, the bottle is lowered into the oil until it reaches the bottom; thus the oil will flow into the bottle while descending, taking a portion of the sample at every level. When the bottom is reached, the bottle is drawn back to the top, keeping it in steady motion, in order to obtain as near an average sample as possible. The operation is repeated until the bottle is filled completely. The rapidity of raising and lowering will depend on the depth of the oil sampled, a greater depth necessitating greater speed in order to cover the whole distance.

If necessary, samples may be taken at any desired depth by attaching another string to a cork fitted loosely in the mouth of the bottle. The bottle is lowered to the point at which the sample is to be taken and the cork then is removed by a sharp pull on the string. Samples also may be taken at different depths by means of an "oil thief" (see page 181); and tanks may be equipped with sample cocks at spaced intervals up their sides, for drawing samples at different levels.

SAMPLING FROM CARS

In sampling cars it is well to take a sample from the bottom, to detect the presence of water or sediment. When cars are being loaded for shipment, four-ounce samples from the car bottoms and an average sample from each car should be retained for future reference in case of controversy. All retained samples should be filed in the order of the dates of shipment.

An average pint or quart sample from each car, properly labeled or tagged, is sent to the laboratory for examination. In case a number of cars are loaded at one time with the same product, it usually is sufficient to determine gravity and color on each car and make an average sample of all the cars by mixing an aliquot portion of each for any other necessary tests. Any variation of gravity or color indicates the desirability of further tests being made on that particular sample.

Samples also may be taken while loading cars by removing an aliquot portion of the oil flowing into the car at spaced intervals of time. These samples may be mixed in a large container, from which is taken the sample to be tested. When a large number of cars are being loaded, a sample from the pump, or stream to the cars, should occasionally be tested to check the tank sample and make sure that the product conforms with the specifications.

WATER AND B.S.

The detection of water in the bottoms of tanks or of cars can be exe uted by several procedures.

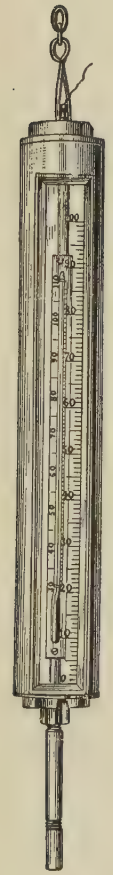

FIG. 73.—An "oil thief."

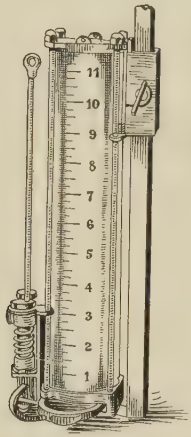

FIG. 74.—Another oil sampling device.

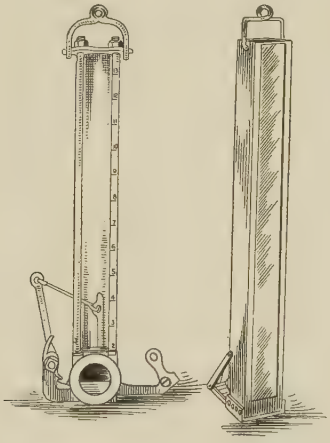

FIG. 75.—Other types of "oil thieves."

FIG. 76.—A type of water indicating apparatus.

The "oil thief" constitutes the basis of a method which is used probably more than any other. The construction of this instrument is that it is equipped with a valve at the bottom which is opened or closed by means of a string, while the top is open; a

tape-line is attached to the "thief," by means of which it is lowered into the oil to the bottom of the tank; and the valve is opened and allows any water to flow into the receiving chamber to whatever level it may be. The "thief" then is drawn up for examination. If the instrument is filled with water, it is lowered again to the bottom and then drawn up to the next level, as indicated by the tape measurement. When the water level is found, the exact amount will be shown on the glass at the side of the "thief.[1]" Figs. 73, 74 and 75 show types of "oil thieves" which now are in use.

Another instrument for indicating the depth of water is illustrated in Fig. 76. This is a square metal tube, approximately 12 in. long and 1 in. wide, with a groove running the full length to hold a strip of sensitized paper. The paper is yellow in color and is changed to white when it comes in contact with water, giving a well-defined line between the oil and the water. Another method consists in coating the surface of a steel tape or gage-pole with chalk; a distinct line of demarcation is shown where the chalk is moistened with water.

It is rather difficult to determine the amount of water and B.S. in tanks containing fuel oil, since portions of the oil and water are emulsified, and there is no defined line of separation between the oil and the water. To determine the amount of water in a tank, samples are taken at each foot above the bottom until the water level is passed. The emulsified samples then are tested for water by means of a centrifuge or by other suitable procedure.[2]

If a sample is worth taking, it should be taken properly, and too much stress cannot be laid on this point.

[1] In addition to operating as described above, this thief is equipped with a rod extending below it, so that the sample is taken automatically at any height from the bottom up to 3 feet. Above this, the valve is operated with a line. On the thieving of petroleum oils, see ELLIOTT, *Oil and Gas J.*, **18** (1920), No. 51, 66.

[2] See page 4.

CHAPTER VIII

THE EVALUATION OF OIL-SHALE[1]

The value of an oil-shale or bituminous shale[2] does not depend entirely upon the yield of crude oil by destructive distillation—that is, upon the content of kerogen,[3] or oil-forming bituminous matter. Of prime importance are the quality and refinability of the crude; hence the nature of the kerogenous substance and the method of distillation which is employed, are factors which largely determine commercial value. Since the yield of ammonia from distillation is of importance, attention must be given to the percentage of nitrogen in the oil-shale.

The *productive value* of an oil-shale property therefore is dependent upon the following considerations:

1. The demonstrated yields of crude oil and ammonium sulphate; that is, the kerogen and nitrogen contents of each bed of oil-shale. This may be expressed as the capacity per unit volume of the property, but, better, the average per seam. Capacity determination can be based only upon representative samples of certain sources, so selected as to demonstrate the variation and continuity in the case of every bed on the property under investigation.

2. The quality or refining value of the crude oil. For the assurance of successful large-scale operation, a retorting process must be used which will give good yields of economically refinable crude-oil and, wherever commercially possible, of ammonia. American oil-shales are comparatively high in characteristic

[1] For a general descriptive account of oil-shale and the shale-oil industry, see Bacon and Hamor, *lib. cit.*, **2**, 807–45; and Gavin, Hill and Perdew, "Notes on the Oil-Shale Industry," Bureau of Mines, 1919.

[2] Reference is not had here to oil-bearing or petroliferous shale, from which crude petroleum may be obtained by mechanical means or by extraction with a solvent.

[3] On the nature of kerogen, see Greene's "A Treatise on British Mineral Oil," 1919, 3 *et seq.*

kerogens, but in the Western States they are generally low in nitrogen-content; while in the eastern Canadian provinces the oil-shale is highly nitrogenous, although it yields an oil which is commercially inferior to the Scottish.

3. The quantity of usable oil-shale in sight. The supply of oil-shale may be extensive and, in fact, practically inexhaustible; but, prior to actually beginning operations, a shale-oil company should be able to claim, say, 50 years' supply of oil-shale affording a minimum net profit of about $1.00 per ton retorted, assuming a throughput of, say, 1,000 tons per day. The property should contain large (especially thick) and accessible seams of oil-shale of uniformly high kerogen and nitrogen contents.[1]

4. Favorableness of the location of the oil-shale field with respect to shipment of products to the markets.[2] It may be noted here that, if physical and economic conditions permit, the plant should be located in the center of the field under development.

The chief factors to be considered in choosing a site for a shale plant are as follows: quality and quantity of shale, proximity to shale beds, mining conditions of shale deposit, water supply, facilities for disposing of spent shale, transportation facilities for the products, labor supply, labor housing, and market conditions. Of these, transportation must be given serious consideration; provision must be made for bringing in the machinery and equipment, some of which is very cumbersome, and for removing the crude shale-oil or the refined products to the market; a plentiful supply of water is absolutely necessary; and if possible the plant should be situated so that the spent shale can be removed easily by gravity.

[1] For an economic consideration of oil-shale, see Bacon and Hamor, *J. Soc. Chem. Ind.*, **38** (1919), 161.

[2] The difficulties that are likely to be encountered in marketing oil-shale products must be given attention. The petroleum industry of this country, established for many years, has developed an extensive and efficient system of marketing its products. Before shale-oil products can become of commercial importance it may be necessary to carry on a campaign of education in order to convince the public that such products can be used with the same degree of satisfaction as those obtained from petroleum. The marketer of petroleum products has a distinct advantage through the largest oil refineries being located either on the seaboard or near the marketing centers, whereas it seems that the shale-oil refineries will be located largely in the Rocky Mountain region.

THE PRELIMINARY EXAMINATION OF OIL-SHALE

Methods Used in Scotland

Crude Oil.—The tubes employed for the determination of the yield of crude oil from a given sample, are made of 2-in. malleable-iron tubing, having a length of 6 ft. and being closed perfectly by a cap at one end. The method of procedure is as follows: A fair and average sample of 1 lb. of the shale in pieces about ½ in. square, and not exceeding about ¼ in. in thickness at any part, is put into the tube, which then is inserted in a furnace for about a foot of its length, inclined at a suitable angle, and heated very gradually to a red heat. No condenser is used in making these tests, so that some of the naphtha is lost, but, by controlling the heat properly,[1] the conditions of destructive distillation are identical with those occurring in a retort of the Pumpherston type. In fact, these tubes were designed with that idea in view, and have been used extensively in Scotland for experimental purposes.

Ammonium Sulphate.—The method used for the determination of the yield of ammonium sulphate obtainable from a sample of shale is known as the Bailey method and is employed in the laboratories of all crude-oil works where Pumpherston retorts are used. It is essentially as follows: Thirty grams of the shale in small pieces are heated in a malleable-iron tube to bright redness in the presence of a current of steam for 1½ hours, the gases resulting being led into a flask containing 2N sulphuric acid. In this solution the ammonia is determined as nitrogen in a nitrometer and the yield of ammonium sulphate is calculated thereby.

[1] The portion of the tube which is within the furnace and which is occupied by the shale is gradually and increasingly heated during a period of six hours, and is finally at a bright red heat. At the end of the distillation no fumes should be visible on looking up the inclined tube and the shale itself should be distinctly and sharply seen lying in the tube. At no stage of the heating should yellow or brown vapors issue from the mouth of the tube, but towards the end a bluish gas may be evolved. When the distillation is complete, the tube gradually is heated *downwards* (by a gas flame) so as to melt the oil which has set in the colder part of the tube (acting as a condenser) and the oil is allowed to drain away into the receiver, which then is covered and carefully put aside for examination.

The crude oil obtained is separated later from the water which accompanies it during the distillation of the shale; it then is measured and its specific gravity is determined as described on page 2. The yield is reported in U. S. gallons per short ton.

The apparatus consists of a 1-in. malleable-iron tube, of ¾ in. internal diameter, and 28 in. in length. Into one end is fitted a cork, through which passes a short brass tube with a stop-cock, the outer end of which is connected with the steam supply. Pieces of previously ignited fire-brick, about 5 mm. in diameter, are dropped into the iron tube so as to occupy about 8 in. of the tube next to the stop-cock. Then 30 g. of the shale, in pieces about 3 mm. in diameter, are placed in the iron tube and the tube is put in a combustion furnace, with the portion containing the shale well in the center of the furnace, so that it may be heated readily to a bright red heat. Into the open end of the tube next to the shale is fitted a cork, through which a

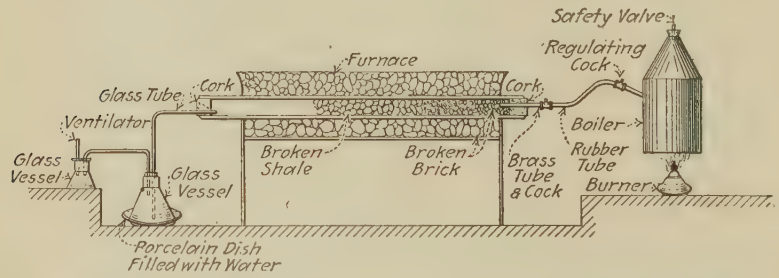

FIG. 77.—Diagram of apparatus used in the determination of the yield of ammonium sulphate from oil-shale, according to Bailey's method.

glass delivery tube passes through a cork into a liter Erlenmeyer flask, the exit tube of which passes to a 200-c.c. flask, which acts as a trap for receiving any liquid which may be carried over from the larger flask; 100 c.c. of 2N acid are introduced into the larger flask before it is connected with the iron tube.

To start the operation, the furnace is lighted, and the iron tube is brought as rapidly as possible to bright redness, the time to attain this being noted (it should not exceed 5 or 6 minutes). As soon as vapors begin to show in the glass tube, the stop-cock is opened cautiously and a regulated and moderate current of steam is allowed to pass through the tube. The proportion of steam should be such that, after 1½ hours heating to bright redness, about 600 c.c. of liquid are contained in the larger flask. During the operation, the large flask should be immersed partly in cold water in a porcelain basin, in which the water is frequently replaced by fresh cold water; and the ends of the iron tube should be kept cold by wet lint or cloth wrapped around

them and kept moist, in order to prevent charring of the corks. After 1½ hours, the apparatus is disconnected, care being taken that neither then nor at any other time does any of the liquid in the flask go back into the tube, owing to reduction in pressure, and the flasks are rinsed out. The filtered liquid is made up to a volume of one liter and then thoroughly mixed by shaking. A measured portion, say 250 c.c., of this liquid then is evaporated in a porcelain dish on a water-bath until its volume is reduced to about 5 or 6 c.c., and this residue is rinsed into the cup of a nitrometer,[1] taking precaution that all the ammonia salts are transferred to the cup. Excess of sodium hypobromite is added, the nitrometer is shaken, and the volume of nitrogen, temperature and pressure are read off with all the necessary corrections, from which data the total volume of nitrogen from the 30 g. of shale is calculated. One c.c. of nitrogen at n.t.p. is equivalent to 0.001-562 g. of ammonia, from which the yield of ammonium sulphate per ton may be calculated.[2] Determinations by this method approximately equal the yields obtained by retorting the shale in a Pumpherston retort, the most satisfactory dual-purpose retort in industrial use.

Alternate Method.—At one shale-oil plant in Scotland, the yield of ammonium sulphate has been determined in the laboratory by collecting the liquid obtained along with the crude oil in a tube test, estimating the amount of ammonia in this, and multiplying by a factor, calculated from the yield of ammonia by a similar shale in a works retort. This method of estimation lacks the nicety of the Bailey method, but is found to be sufficiently accurate for some purposes.

APPARATUS FOR FIELD DISTILLATION

The portable distilling apparatus used by the United States Geological Survey in its field investigations in Northwestern Colo-

[1] The nitrometer is employed in determining the amount of ammonia absorbed by the sulphuric acid, since various basic substances distil over which would render a determination by titration inaccurate.

[2] Calculation:

Example.—30 g. of shale.

Liquid made up to 1000 c.c.

250 c.c. in nitrometer gave 24.67 c.c. of nitrogen at N.T.P.

Then 24.67 × 4 = 98.68 c.c. of nitrogen from 30 g. of shale, and 98.68 × factor 0.4524 = 44.64 lb. of pure ammonium sulphate per long ton. And 44.64 × factor 1.03 = 45.98 lb. of commercial ammonium sulphate (containing 25 per cent. of ammonia) per long ton.

rado and adjacent areas, is applicable for the conduct of laboratory assays, although the Scotch methods are more satisfactory for work other than in the field.[1] The Geological Survey apparatus consists of the following essential parts:

Two gasoline blast lamps (Barthel's).
One iron mercury retort (one-half pint), with delivery tubes.
One brass condenser.
Two ring stands.
One 3½-in. ring.
One large condenser clamp.
One receiver for condensed liquids (50 c.c. glass graduate).
One ammonia scrubber (8-ounce bottle filled with glass beads).
Two pairs of combination pliers.
One postal balance.
Six ft. of rubber tubing.
Glass tube for connecting condenser, receiver and ammonia scrubber.
One glass separatory funnel.

Because of its simplicity and because its flame can be adjusted to any desired angle or length, the Barthel blast lamp was chosen to furnish heat for the still or retort. This lamp consists of a small spherical gasoline tank with burner, mounted on an iron base in such a way that the burner may be turned to any angle. To manipulate the burner, the tank first is filled nearly full of gasoline and the cap is screwed down securely. Gasoline is placed in the small cuplike depression around the burner and lighted. When this gasoline is burned out, sufficient heat will have been produced to generate gas under pressure, which may be lighted at the end of the burner on opening the burner valve. If the flame is yellow or sputters, the burner is not sufficiently hot and must be reheated. The gasoline tank of each burner holds sufficient fuel to keep the blast burning about 2 hours. Inasmuch as each distillation of shale requires from 3 to 4 hours, two burners are used with each retort. The second torch may be lighted most easily by playing the flame of the first on the burner of the second.

The vessel in which the shale is placed to be heated is an ordinary iron mercury retort (capacity, one-half pint), which is equipped with close-fitted lid and clamp and an iron delivery-tube. The delivery-tube is fastened to the inner tube of the con-

[1] Field tests as used are inadequate for indicating the best methods of retorting. Laboratory tests are more dependable, but properly planned experimental plant operations afford the most reliable results.

denser by a small brass plumber's union, which provides a very easily disconnected joint. The retort is held in place above the flame of the burner by a ring and ring-stand. The condenser used in the outfit consists of an inner tube of thin brass, three-eighths of an inch in diameter and 15 in. long, to one end of which is soldered a small brass plumber's union. The inner tube is surrounded by a second thin-walled brass tube, 1¼ in. in diameter and 11 in. long, which is provided with a two-hole rubber stopper at each end, one hole being for the inner tube. A small brass

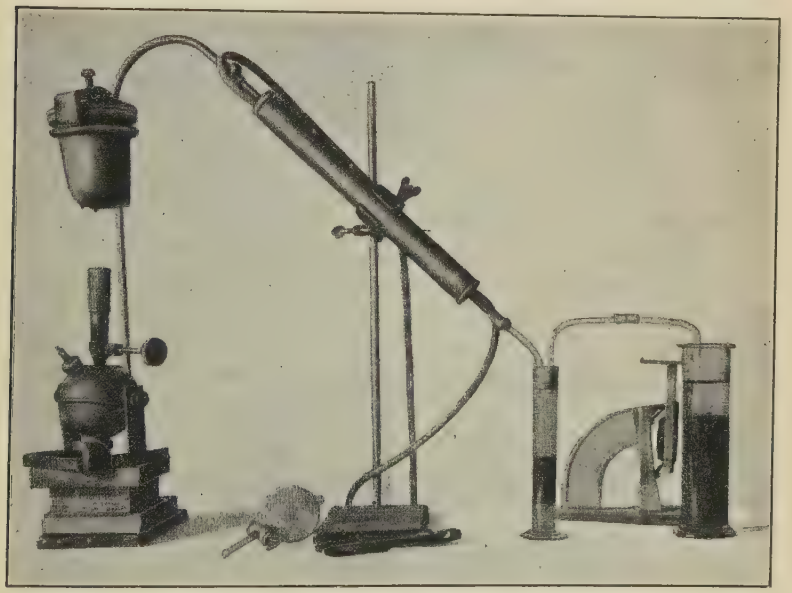

FIG. 78.—The apparatus for conducting distillation-tests on oil-shale inthe field.

tube, 2 in. long, is inserted through the second hole of each stopper to provide connection for the entrance and waste of the water which is circulated between the inner and outer tubes to keep the inner tube cool. The delivery tube from the retort is so bent that when the retort is in an upright position the condenser will be turned at an angle of about 40° from the horizontal. The condenser is held in position by a single clamp, attached to a ring-stand. The receiver for the condensable products of the distillation consists of a 50-c.c. glass graduate, provided with a two-hole rubber stopper through which are thrust two glass tubes, one for the entrance of the liquids and permanent gases from the

condenser, and the other for the escape of the permanent gases to the ammonia scrubber. The glass tubes have a diameter of about ¼ in. and are bent at the proper angle to make connections with the condenser and scrubber. The tubes should barely penetrate the cork. The ammonia scrubber consists of an ordinary glass cylinder or 8-ounce wide-mouthed bottle, provided with a glass tube reaching nearly to the bottom of the bottle for the entrance of permanent gas from the receiver. The bottle is filled with glass beads, which provide additional surface and a means of breaking up the gas into small bubbles as it passes up through a 15 per cent. solution of sulphuric acid. The water for cooling the condenser may be had from waterworks, or, if no running water is at hand, a tank or tube may be stationed near the condenser, at a slightly higher level, and the water conveyed over the top by a siphon entering the condenser at the lower end and wasting at the upper end. The pliers are used for handling the retort, while the postal balance has a capacity of 4 pounds and is used to weigh the sample of shale.

In order to determine the quantity of oil and ammonia that may be derived from a sample of shale, the shale first is broken up to pass through a screen of ½-in. mesh. After thorough mixing, a sample weighing 8½ ounces is so selected as to represent the entire quantity. This sample is placed in the iron retort and the cover is fastened securely. In order to prevent leaks, the joint between the cover and retort bowl is plastered with a thick paste made of a mixture of powdered graphite and glycerin. The delivery tube from the retort then is coupled with the inner tube of the condenser. The ammonia scrubber is filled approximately two-thirds full with a 15 per cent. solution of sulphuric acid, and cool water (not ice water) is started circulating through the condenser. The blast lamp then is lighted and placed beneath the retort, with the flame turned as low as possible. After heating about ten minutes, water and oil will begin to condense and be delivered into the receiver. The permanent gas will pass into the ammonia scrubber and bubble up through the sulphuric acid, which will combine with any ammonia contained in it, producing soluble ammonium sulphate. Gentle heat should be applied to the retort as long as any oil is delivered to the receiver; then the flame of the burner may be lengthened until, at the end of three or four hours, the burner will be at full blast, the retort will be red hot, and the shale will cease to yield either

oil or gas. The products of the distillation then are measured; and the quantity of oil in the receiver is recorded, as well as the quantity of water in the same receiver. The liquid products of the distillation then are transferred to the separatory funnel, and the water is drawn off from the oil and added to the liquid contained in the ammonia scrubber. The material in the ammonia scrubber then is placed in a glass-stoppered bottle and transferred to the laboratory for the determination of the quantity of ammonium sulphate.[1] The yield of oil in United States gallons to the short ton of shale is equal to the number of cubic centimeters of oil in the receiver, provided the sample of shale used weighed 8½ ounces. The oil obtained from the distillation should be placed in a small bottle for determination of its specific gravity, which can best be done in the laboratory. In order to compute the number of pounds of ammonium sulphate which may be derived from a short ton of shale, it is necessary only to multiply the number of grams of ammonium sulphate found in the sample by 8.8.

CRUDE OIL YIELDS OF AMERICAN SHALES

The following figures are based on the results of one hundred and thirty-two analyses published by the United States Geological Survey, forty-two analyses made in the laboratories of the Colorado School of Mines, and thirty-one analyses from other sources. Fifty-four of the analyses are on Colorado shales, fifty-two on Utah shales, forty-five on Wyoming shales, and fifty-four on Nevada shales.

[1] The average amount of ammonium sulphate produced from shale by steam distillation is about two and one-half times the amount obtained from the same samples by dry distillation, thus providing a factor for the conversion of the figure for ammonium sulphate by dry distillation to ammonium sulphate which may be obtained with steam distillation (the method practiced in the shale-oil industry of Scotland and France).

In six samples tested, an average of 37.8 per cent. of the nitrogen in the shale was accounted for in the ammonium sulphate obtained by steam distillation, compared with an average of 15.7 per cent. recovered by dry distillation. The yield of ammonium sulphate was determined for 57 of the samples that yielded more than 15 gallons of oil to the ton of shale. In these samples an average of 6.7 pounds of ammonium sulphate to the ton was obtained. This multiplied by 2.5, the factor mentioned above, gives an average of 16.7 pounds of ammonium sulphate to the ton, which seems to be a fair estimate of the quantity that may be produced in commercial practice from shale of the area examined in 1914 and 1915 by the Geological Survey (see *Bull.* **641-F**, U. S. Geol. Surv., 1916).

No. of analyses		Unit	Minimum	Average	Maximum
205	Shale oil	Gallons per ton*	0.3	38.0	90.0
205	Shale oil	Specific gravity	0.832	0.890	0.950
163	Ammonium sulphate	Pounds per ton*	0.4	9.4	20.0
64	Gas	Cubic feet per ton*	400.0	3800.0	5600.0
26	Water	Gallons per ton*	2.0	4.8	8.5
26	Spent shale	Pounds per ton*	900.0	1200.0	1800.0
26	Sulphur	Per cent	0.25	0.80	5.20
16	Heating value	B.t.u	1000.0	4500.0	8000.0
6	Carbon	Per cent	0.83	22.5	37.2

* Short-ton.

Shale distillations with steam yield a few more gallons of oil a ton than dry distillations and the specific gravity of the oil is between 0.03 and 0.04 greater. Steam distillations also increase the quantity of ammonium sulphate about two and one-half times the value obtained by dry distillation. The values given in the table above were obtained by dry distillation.

Until some standard method and apparatus are adopted, comparison of the work of different analysts is extremely difficult, if not impossible. Plant results would probably differ considerably from any of the yields obtained by the laboratory methods now in use.

THE METHOD IN USE AT THE MELLON INSTITUTE OF INDUSTRIAL RESEARCH FOR THE ASSAY OF OIL-SHALES FOR YIELD OF CRUDE OIL[1]

The apparatus used at the Mellon Institute in assaying oil-shales for yield of crude oil, is shown in Fig. 79. *R*, the retort, is a piece of 1½-in. wrought-iron pipe, capped at both ends. It is heated by a battery of six bunsen burners with wing tops. The condenser consists of a piece of ⅛-in. pipe provided with an 18-in. Liebig condenser near its lower end. The retort is tapped at *A*, and a tube is provided running through the entire length of the retort, and lying close to the bottom of the mass of shale. By the use of a copper-constantan thermocouple inserted to various lengths, the temperature may be obtained at any point along the retort, and the whole mass of shale kept at a uniform temperature.

In making a run, a charge of from 250 to 300 g. of the shale is placed in the retort. This may be a sample obtained by the usual methods of sampling, but it is procured preferably by sepa-

[1] This method was worked out by C. L. Jones, while research assistant to Director R. F. Bacon.

rating the original sample into particles under and over 10-mesh, and recombining these in their original proportions to obtain the proper weight of sample. The iron condenser-tube is weighed before setting up the apparatus.

In heating the retort, the flame is not allowed to come into contact with it. The rise in temperature may be controlled by the pyrometer; but if care is taken to heat the retort very gradually, this is not necessary to obtain consistent results. The tip of the flame should be ¾ to 1 in. from the retort. Heating is continued in this way for four hours, or until no more distillate is secured. The flame then is raised gradually until the tip of the flame just touches the retort. After continuing this for about three hours more, the flame is turned up and the distillation is finished at a red heat. At the end of this time the glass water-jacket is removed, and the condenser tube is unscrewed from the retort and weighed. As a check, the retort itself may be weighed before and after the run. The weight of the distillate in the receiver having been obtained, a figure then is had for the total oil, water, and dirt in the condenser and the receiver. As much as possible of the oil in the condenser then is drained into the receiver by warming the tube. Gasoline or petroleum ether in successive portions is added and decanted until only the water remains in the receiver. This is allowed to stand at room temperature until the petroleum ether odor disappears, and the water is weighed.

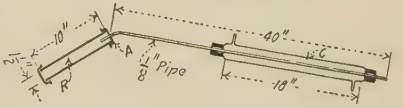

FIG. 79.—The apparatus used at the Mellon Institute of Industrial Research for assay of oil-shales for yield of crude oil.

Results obtained for oil yield check within 0.5 gal. per ton. When runs are to be made at specific temperatures with pyrometric control, the retort is enclosed in an air jacket of 3-in. pipe. Due to the extreme simplicity of the apparatus it is relatively easy to secure the gradual and uniform heating essential to obtain the highest yield. Higher yields and more consistent results were obtained with it than with the mercury retort apparatus described above. It is also easier to maintain a uniform temperature in the laboratory with this form than with a vertical tube retort. For steam distillation the cap on the lower end of the tube is replaced with a tube through which steam is passed. For this purpose the apparatus is probably not so satisfactory as the

vertical form of retort. A means of measuring the gas given off may be added when desired.

The chief sources of inaccuracy in laboratory assays seem to be:

1. Leaky retorts.

2. Retorts the size and shape of which make uniform and gradual heating difficult, and which afford cool zones which run a part of the oil back into hotter zones. The mercury retort form is an example of this.

3. The attempt to distil larger charges of oil-shale than the apparatus can heat uniformly throughout.

4. The considerable error introduced by the amount of oil in the condenser, since but a small charge is used.

THE EXAMINATION OF CRUDE SHALE-OIL

For the purpose of determining the yield of products from the crude-oil of a specific oil-shale, the destructive distillation of the latter is carried out preferably in a suitable retort,[1] and the apparatus described above may be used provided a roughly approximate refining value is desired. The crude product then is examined in accordance with the procedures recommended for crude petroleum (see page 1). In this way the commercial value of the crude oil produced by any distillation process under investigation may be found and reported upon, as described on page 22. In particular, it should be ascertained whether the crude should be refined *in toto*, or whether it should be merely skimmed.

Methods Used in Scotland

The following is an outline of the schemes used by Scottish experts[2] for refining crude shale-oil in the laboratory, in order to ascertain the yield of commercial products:

[1] For the description of a laboratory retort used by Baskerville and Hamor, see *J. Ind. Eng. Chem.*, **1** (1909), No. 8, 507. On a horizontal gas-fired retort for conducting the destructive distillation of bituminous substances, see Ingram, *Bull. School of Mines and Met., Univ. Mo.*, **3**, 65 pp. (1917). The fuel-testing laboratory of the Mines Branch of the Department of Mines of Canada uses the apparatus shown in Fig. 80 for destructive-distillation tests on oil-shales. See also Leverin, *Rept.* **59**, Mines Branch, Canada Dept. of Mines. On the testing of oil-shales, see finally Nicolardot and Baurier, *Chim. et Ind.*, **2** (1919), 770 and 777.

[2] For another method, see that of Tervet, described in Redwood's "Petroleum and Its Products," **2**, 644; and in Allen's "Commercial Organic Analysis," 1907, **2**, ii, 39.

Method A.—The crude oil is distilled and fractionated into "crude naphtha" (1) and "crude distillate" (2). The latter is treated with sulphuric acid (1.84 sp. gr.) and caustic soda solution (1.35 sp. gr.), and again distilled, fractioning into "crude burning oil" (3), "heavy oil" (4), and "residuum" (5). Products Nos. 1 and 3 are refined chemically by treatment with sulphuric acid, followed by distillation over caustic soda. No. 4 is cooled to a low temperature and filter-pressed to recover

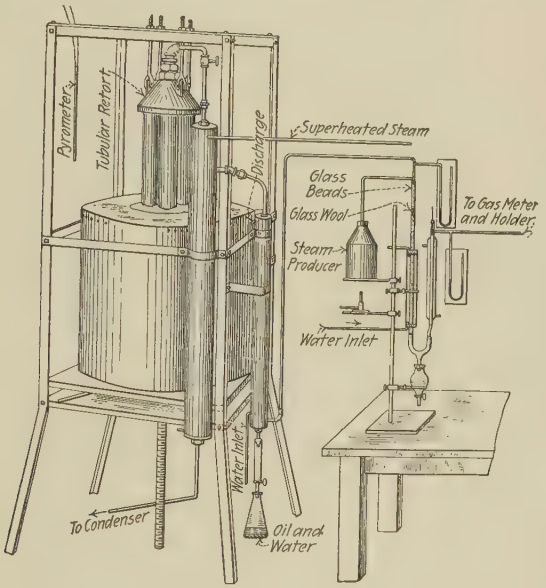

FIG. 80.—Apparatus for the destructive distillation of lignite and bituminous minerals.

The method employed is as follows: The hot gases leaving the retort pass down through the center tube of a small scrubber made of iron pipe and containing three interlacing coils of wire, passing up again through a surrounding annular space; the whole being jacketed with superheated steam. The heavy tar oils are here condensed in a practically water-free condition, and drop into a weighed glass receiver. The lighter oils, steam, and gases pass on and down through a simple tubular condenser, where the two former condense and collect in a receiver, the oils floating on the water and showing only a slight tendency to emulsify. The cool gases leaving the condenser still contain some tar-fog; they are therefore passed down through a tube scrubber filled with glass beads (and a short layer of glass wool), through which a jet of steam from a weighed boiler is also passed. The bottom half of this scrubber is water-cooled. This scrubber completely removes the tar-fog from the gas; the oil first condensed on the beads acts as an oil-scrubber, collecting more of the tar; the steam prevents the clogging of the scrubber by keeping the tar hot and fluid, and also, when condensing at the bottom, carries down with it any vapors still remaining. The gases are thus completely cleaned, and all the liquid products as well as the ammonia from the lignite are collected in the vessels and can be readily weighed and examined. The tar oils thus collected are reasonably free from water and can be redistilled without excessive bumping or frothing. The gases leaving the scrubber pass through a final cooling tube, through a gas meter, and into a gas holder.

solid paraffin (6). The "blue oil" (7), obtained from (4), is treated with sulphuric acid of 1.72 and 1.84 sp. gr., and then a

soda solution of 1.35 sp. gr. Next it is distilled over solid caustic soda, and fractioned into lubricating oils, which then are refined.

Method B.—The crude oil is treated with sulphuric acid (1.72 sp. gr.), and then distilled and fractioned into "crude naphtha" (1) and "crude distillate" (2). The latter is treated with sulphuric acid of 1.72 and 1.84 sp. gr., and again distilled, fractioning into "crude burning oil" (3), "heavy oil" (4), and "residuum" (5). No. 4. is cooled and filtered, giving solid paraffin

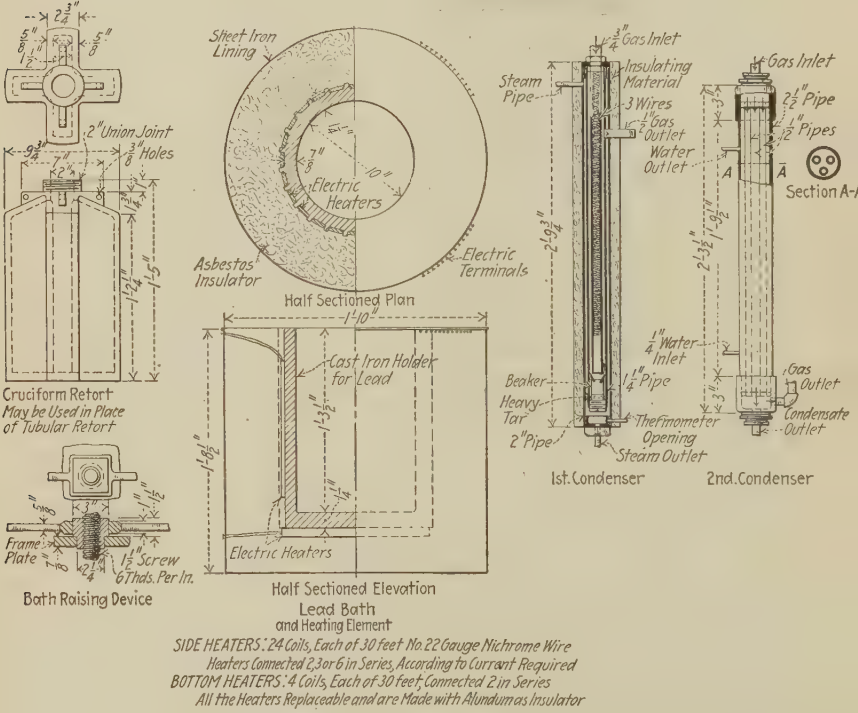

Fig. 81.—Details of lignite distillation apparatus.

(6) and "blue oil" (7). The latter is treated with sulphuric acid (1.84 sp. gr.) and caustic soda, distilled off solid caustic soda, and fractioned into lubricating oils.

All the distillations are conducted with external heat and with the use of a current of steam, except the distillation of the "crude naphtha," in which only steam is employed. The quantity of steam used is usually such that the condensed "steam water" is equal in volume to about 30 per cent. of the oil distill te.

The only exception to this is in the distillation of the "heavy oil," when the steam is shut off and the external heat is increased so as to cause cracking of the "still-bottoms," with the production of crystalline paraffin, but the steam is turned on to carry over the last of the "bottoms."

METHOD OF REPORTING RESULTS

It is appropriate to present in conclusion an outline of the procedure which usually is followed in reporting the results of a general examination of an oil-shale.

The statement regarding the preliminary examination should include the results of the following determinations: specific gravity, moisture, ash, volatile matter, fixed carbon, sulphur, and nitrogen.[1]

Then follow the results obtained by application of the Scottish tube test (yield of crude oil) and the Bailey method (yield of ammonium sulphate). In addition, the oil-shale should be assayed for crude-oil yield according to the method described on page 192, and the result given for comparative purposes. The yield of crude oil is reported in U. S. gallons per short ton of shale, and the specific gravity and Baumé equivalent of the oil also are stated. The yield of ammonium sulphate is presented in pounds per short ton of shale, both for the pure salt and for the commercial product (containing 25 per cent. of ammonia).

When a particular distillation process is under investigation or the oil-shale is retorted in an experimental plant, a log is kept of the quantity of shale used daily; the total number of gallons of crude oil produced, its specific gravity, and the average yield in gallons per ton; and the production of ammonium sulphate in pounds. At the conclusion of the run, the quantity of shale put through during the test is totalled, as are the gallons of crude oil and pounds of ammonium sulphate produced; the average yield in U. S. gallons per short ton of shale and the average specific gravity of the crude are determined and stated, and there also is given the average yield of both pure and commercial ammonium

[1] These analytical determinations should be conducted according to the methods recommended for coal analysis by the joint Committee of the American Society for Testing Materials and the American Chemical Society, described in *J. Ind. Eng. Chem.*, **9** (1917), 100.

sulphate in pounds per short ton of shale. An average analysis of the uncondensed gas, after leaving the ammonia scrubber, is reported, and also the calorific value and specific gravity of the gas generally are included in the summary.

The results of the evaluation of the crude oil, carried out as described, are presented as in the case of a crude petroleum.

CHAPTER IX

LABORATORY METHODS FOR BENZOL-RECOVERY PLANT OPERATION

By F. W. Sperr, Jr.[1]

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The importance of the benzol industry has achieved general recognition during the last five years. The recovery and rational utilization of as large amounts as possible of benzene and toluene, which are necessary in the manufacture of important explosives and chemicals and of value in the preparation of certain motor fuels,[2] must be considered a national necessity. There are two indispensable steps in securing the most efficient conservation of our benzol resources, namely:

[1] Chief Chemist, The Koppers Company, Pittsburgh, Pa.; and Advisory Fellow, Industrial Fellowship on Coke Technology, Mellon Institute of Industrial Research of the University of Pittsburgh.

[2] See page 57.

1. The installation of the most efficient type of benzol-recovery plant wherever sufficient benzol is being produced to warrant recovery.

2. Careful control of the operation of each plant so as to reduce losses to a minimum.

The second step involves the use of correct laboratory methods for the testing and analysis of the various materials and products. The importance of this phase of the industry must be emphasized highly, and in view of this the author has obtained the consent of The Koppers Company to publish some of the laboratory methods that it has found valuable. These cover all the ordinary requirements of the benzol plant operator and chemist, for whose assistance, principally, the somewhat detailed descriptions have been prepared.[1]

In studying the methods from an operating standpoint, it will be of assistance to the reader to review briefly the method of operation of the benzol-recovery plant. The Koppers system, which is the most generally used in America, is shown on the accompanying diagram, Fig. 82.

The gas is brought to the proper temperature in the cooler *a*, and then passes into the washer *c*, where it is treated with an absorbent oil, which scrubs out all the benzols. The enriched wash-oil is pumped from the scrubbers to heat exchangers *e* and *f*, which are heated by the benzol vapors and by the hot debenzolized wash-oil, respectively. It then is heated still further to the maximum temperature desired by means of live steam in the superheater *g*, from which it passes into the still *h*. In this still the benzols are expelled entirely in the form of vapor. This hot vapor gives up part of its heat to the incoming wash-oil in the heat exchanger *e*, and then is condensed and separated from water in cooler *i* and separator *j*. The hot wash-oil leaving the bottom of the still passes through heat exchanger *f*, where it gives up part of its heat to the incoming wash-oil, and then is finally cooled in water cooler *k*. The cool oil is delivered to circulating tank *d*, from which it is pumped back again over the scrubbers; thus completing the cycle. The light oil is distilled in crude still *l*, and the various fractions are purified by treatment with

[1]For the analytical methods of The Barrett Company, see the following papers by J. M. Weiss: crude tars, *J. Ind. Eng. Chem.*, **10** (1918), 732; distilled tars and pitches, *ibid.*, 817; heavy and middle oils, *ibid.*, 911; and benzols and light oil, *ibid.*, 1006.

acid and soda in agitator *o*, and by rectification in the pure still *r*.

The routine tests usually required in the operation of a benzol-

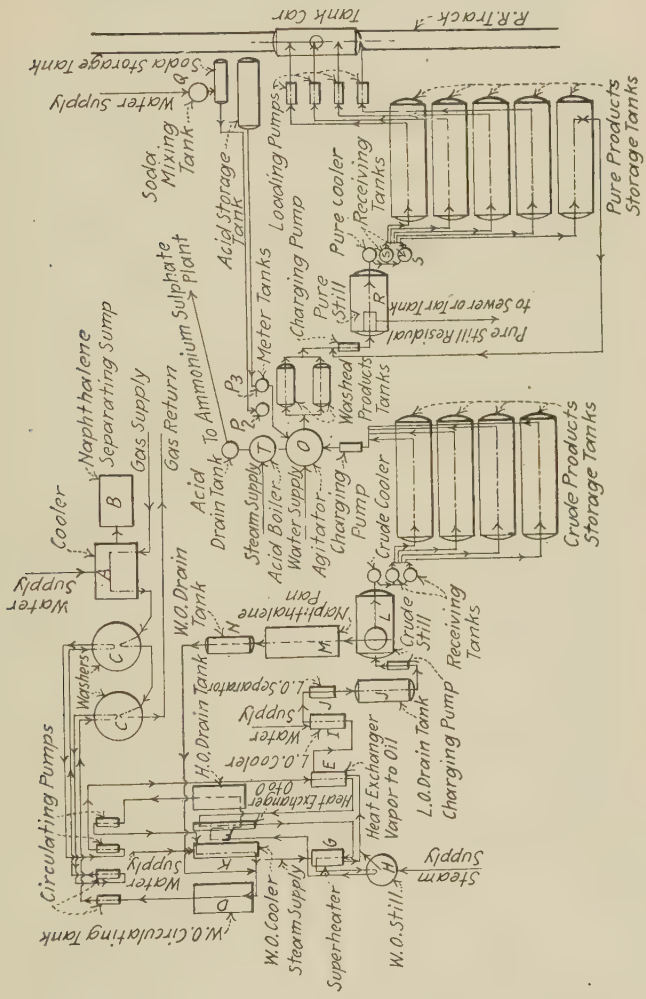

FIG. 82.—Diagram of the Koppers system of benzol recovery.

recovery plant, completely equipped for the production of pure benzene and toluene, may be classified as follows:—

(A) TESTS OF GAS:

1. Determination of light oil in gas entering scrubbers.
2. Determination of light oil in gas leaving scrubbers.

(B) Tests for Wash-Oil Still Operation:

1. Determination of light oil in benzolized wash-oil.
2. Determination of light oil in debenzolized wash-oil.
3. Tests of the light oil:
   (*a*) Boiling-point.
   (*b*) Determination of wash-oil.

(C) Tests for Crude Still Operation:

1. Receiver tests; *i.e.*, boiling-point tests made to control fractionation.
2. Boiling-point tests of fractions (crude benzol, crude toluol, etc.) and residues sampled from their respective storage tanks.

(D) Tests for Agitator Operation:

1. Distillation and acid tests of washed benzols.
2. Tests for SO_2 in washed benzols.
3. Specific gravity of regenerated sulphuric acid.

(E) Tests for Pure Still Operation:

1. Receiver tests; *i.e.*, boiling-point tests made to control fractionation:
   (*a*) Boiling-point.
   (*b*) Acid test.
2. Tests of pure products sampled from storage or running tanks or from shipments:
   (*a*) Boiling-point.
   (*b*) Acid test.
   (*c*) Specific gravity.
   (*d*) Freezing-point (occasional in case of benzene).
3. Boiling-point tests of still residues.

(F) Tests of Materials Used in Operation:

1. Wash-oil:
   (*a*) Specific gravity
   (*b*) Viscosity.
   (*c*) Emulsification.
   (*d*) Cold test.
   (*e*) Distillation.
   (*f*) Olefines.
2. Sulphuric acid—Specific gravity.
3. Soda—Na_2O.

METHOD FOR THE DETERMINATION OF LIGHT OIL IN GAS

Apparatus.—1½-in. steam-jacketed sampling pipe with valve (Fig. 83).

11 reagent bottles, 16-oz., narrow neck, with rubber stoppers.
1 wet gas test-meter reading to 0.001 cu. ft., with thermometer.
1 steam distilling apparatus, 1-gallon capacity.
2 Liebig condensers—15 in.
1 separatory funnel—300-c.c.
1 special distilling flask—200-c.c.
1 measuring tube, 50-c.c., graduated in 1/10th c.c.
1 thermometer—200°C.
1 Hempel dephlegmating column (Fig. 84).

Burners, supports, and tubing, to complete apparatus as described below.

The *method* consists of three steps, viz.:

1. Extraction of the light oil with wash-oil.
2. Steam distillation of the benzolized wash-oil.
3. Dry distillation of the separated light oil.

The wash-oil must be a petroleum product corresponding to specifications given on page 232.[1]

Referring to Fig. 83, *B* is a ½-in. sampling pipe inserted in the gas main (*A*). The end in the center of the main is bent in the direction of the flow of the gas. In case the gas contains much

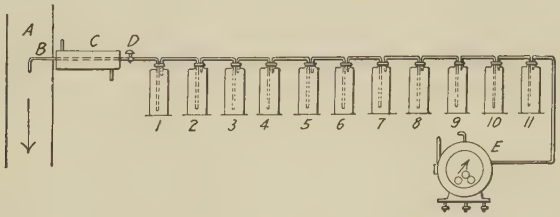

FIG. 83.—Absorption apparatus for light oil in gas.

naphthalene, provision must be made to heat a portion of the sampling tube where it emerges from the gas main. This may be accomplished as shown in the figure by means of a steam jacket about 20 in. long and 2 in. in diameter. When the naphthalene is in large quantity, causing stoppage, it may be necessary to keep the first two or three bottles of the absorbing train immersed in warm water, and to wrap all connections with asbestos or flan-

[1] A complete blank test must be run on this wash-oil.

nel. The flow of the gas is controlled by means of the valve (*D*). The ends of the rubber stoppers that would be exposed to the gas are covered with sodium silicate solution just previous to each test. If this precaution is not taken, the rubber will absorb appreciable quantities of benzol.

Bottle No. 1[1] contains about 100 c.c. of sodium hydroxide (1 part NaOH to two parts H_2O). Each of the other ten bottles contains about 150 c.c. of wash-oil. Rubber tubing is used to connect up the train, but the ends of the glass tubes must be brought closely together so that as little as possible of the rubber may be exposed to the gas. The wash oil must be maintained at a temperature not to exceed 30°C. throughout the test. If necessary, the bottles must be placed in a trough through which cold water is circulated, or else the water may be kept sufficiently cold by adding pieces of ice from time to time.

The gas passing from the train is measured by means of the wet test-meter, which must be provided with a thermometer, so that readings may be taken for temperature correction. As a rule, this type of meter is not suitable for measuring over 100 cu. ft. of gas, and when gas containing very small amounts of light oil is tested, it is advisable to use a somewhat less sensitive meter of larger capacity. The water in the meter in all cases should be saturated with the gas, and should be at the proper level.

Having connected up the train as shown, heat the sampling tube and let the gas blow out of the valve *D* until the tube is well cleared. Then connect bottle No. 1 with the valve, turn on the gas and inspect the train carefully for leaks. It is usually necessary to have the stoppers securely wired down. Now adjust the rate of flow.[2] Note and record the meter temperature and the barometric pressure every hour during the test and adjust the rate of flow as often as may be necessary.

The amount of gas to be scrubbed depends on its light-oil content. When testing gas before the benzol scrubbers, it is best to regulate the gas volume so that not more than 35 or less than 20 c.c. of light oil will be obtained. In the case of debenzolized gas sampled after passing the scrubbers, it is necessary to take about 100 cu. ft. in 24 hours.

[1] In testing unpurified water-gas, a glass tower filled with oxide and shavings should be substituted for this bottle.

[2] This rate should not exceed 2 cu. ft. per hour for benzolized gas, or 4 cu. ft. for debenzolized gas.

When the required volume of gas has passed through the apparatus, close the valve *D* and disconnect the individual bottles of the train. Pour the wash oil into the copper still of the steam distillaton apparatus and rinse each bottle and dip tube with a little fresh wash oil, pouring the rinsings also into the same still. The train now may be filled and connected up for the next test.

Steam Distillation.—Part of the apparatus for this process is shown in Fig. 84. There is a copper still of 1 gallon capacity, fitted with a screw cap through which pass the inlet tube for the the steam, the exit tube to the dephlegmating column, and the thermometer. The thermometer bulb must dip below the surface of the wash-oil and the 130°C. mark must be visible outside the still. The dephlegmating column is of brass and preferably is wrapped in asbestos to prevent undue condensation. Dimensions for this are given on the drawing; the bead column is about 6 in. long. The inlet tube is connected with a suitable source of dry steam by means of a short piece of rubber tubing. In default of a regular supply of boiler steam, an ordinary 1-gallon can, fitted with the usual rubber stopper, safety tube and exit tube, and heated by means of a large burner, makes a very satisfactory steam generator. The neck of the dephlegmating column, which has been bent over, is connected with a 15-in. Liebig condenser. The lower end of the condenser tube passes into a 300-c.c. separatory funnel. Corks must be used for all connections that will be exposed to light-oil vapors. The separatory funnel is wrapped with a cotton cloth, and the outlet tube of the condenser is arranged so that cold water will run over this cloth and keep it thoroughly drenched. A small piece of cotton wool is placed in the mouth of the separatory funnel and around the end of the condenser to prevent currents of air causing undue loss. The separatory funnel is graduated by putting in 25 c.c. of water, marking the level, and then adding 100 c.c. more and marking again.

Place a burner under the copper still and heat until the thermometer shows a temperature of 140°C. Now turn on the steam and adjust the burner so that the temperature does not exceed 155°C. nor fall below 145°C. Water and oil will collect in the separatory funnel. When the water-level reaches the 125-c.c. mark, run out 100 c.c. Repeat this until 500 c.c. have been run out, at which point the distillation may be considered complete.

Shut off the steam and gas and remove the separatory funnel from its support. Run the remainder of the water out carefully. Add to the contents of the funnel 10 to 20 c.c. of a saturated solution of calcium chloride and shake well. Let stand until clear and then run off the calcium chloride solution. Wipe the outlet tube of the funnel dry with a little cotton.

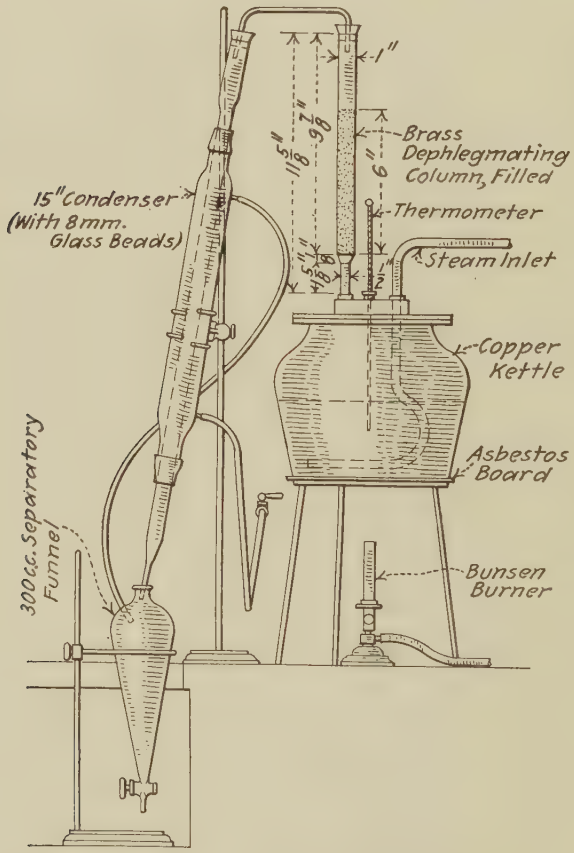

Fig. 84.—Steam distillation apparatus.

Dry Distillation.—Run the dried oil from the funnel into the special distilling flask (Fig. 85) fitted with a cork stopper and thermometer, with a 5½-in. column of 6-mm. glass beads, and connected with a 15-in. Liebig condenser or a standard Barrett condenser. A 50-c.c. measuring tube is placed so as to catch

the distillate. Heat the contents of the distilling flask with a small flame, until the vapor temperature reaches 200°C. Allow the condenser to drain a few minutes, then read the volume of oil distilled, correcting for water if present.

Calculations.—Average the barometric pressures and the meter temperatures for the period of the test. Correct the observed gas volume to 760 mm. pressure and 15°C. with the aid of the usual formulas or tables.

A = corrected gas volume.
B = cubic centimeters of light oil obtained.

Then: gallons of light oil per 1000 cu. ft. of gas $= \frac{0.264 \times B}{A}$.

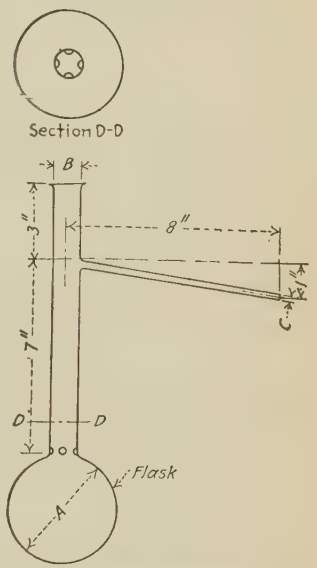

FIG. 85.—Flask for fractionation.

It will be noted that the result is not expressed as the light-oil content of the original gas, but refers to the gas as measured after the process of extraction. This is in accordance with operating conditions, since it is the general practice to meter the gas after it passes the benzol scrubbers. It is hardly worth while making correction for CO_2 and H_2S removed by the sodium hydroxide in the test apparatus, except in cases where these two constituents may be unusually high.

NOTES.—(1) The number of bottles in the absorbing train has been worked out by experiment and cannot be reduced without risk of a loss of light oil.

(2) An automatic siphon arrangement may be used for running off the water from the steam distillation, dispensing with watching the separatory funnel. The volume of water in this case should not be reduced much below 75 c.c.

THE ANALYSIS OF LIGHT OIL

The following method was devised for the purpose of estimating the percentage of benzene, toluene, and solvent naphtha in the light oil obtained in connection with the above described method for the determination of light oil in gas.

Ordinarily the light oil produced from two or three tests may be combined for each analysis. The method has been employed for some time and has given good results for the usual run of light-oil samples, which generally contain at least 50 per cent. of benzene and not over 30 per cent. of toluene. The accuracy of the method is much less for light oil containing less benzene or more toluene, but such material is not encountered so often. If necessary, the simple expedient can be adopted of adding sufficient pure benzene to bring the composition of the sample within the proper limits. Such check determinations as have been made on samples of known composition show that, with careful attention to details, the amounts of benzene, toluene and solvent naphtha can be determined, with an error in each case amounting to about 1 per cent. of the total light oil, which is sufficiently satisfactory for ordinary purposes.

Purification of the Light Oil.—An adequate quantity of the light oil is measured from a burette and distilled to 200°C., using a flask fitted with a Hempel column, containing 5 or 6 in. of glass beads. In the testing of samples produced in connection with the determination of light oil in gases this preliminary distillation is made as part of such determination, and need not be repeated. It may be omitted in other cases at the discretion of the chemist. The distillate is caught in a suitable burette and measured. A quantity—preferably about 100 c.c., and in any case not less than 50 c.c.—is drawn off into a slender 300-c.c. separatory funnel, and treated with 6 per cent. of its volume of concentrated sulphuric acid (sp. gr., 1.84). This treatment is in two stages.

First, two-thirds of the sulphuric acid is added, then one-third. After each addition of the acid, the mixture is shaken about five minutes, allowed to stand until separation is complete (about 15 minutes), and the sludge drawn off. The light oil now is washed with lukewarm water (about 40°C.) and this is drawn off carefully. Then 5 per cent. of its volume of sodium hydroxide solution (sp. gr., 1.107) is added; the mixture is shaken for 5 minutes, allowed to settle about 15 minutes or until clear, and the alkali solution carefully drawn off. Finally, the oil is washed with warm water (about 40°C.) and this is drawn off.

Most of the water is removed by washing with 15 to 20 c.c. of saturated calcium chloride solution. After this has been drawn off, a little solid $CaCl_2$ is added and the mixture allowed

to stand over night. The light oil then is decanted back into the burette and measured. The difference in volume gives an indication of the loss to be expected by sulphuric acid purification on a large scale. The specific gravity of the washed light oil is determined (at 15°C.), using a pycnometer or Westphal balance.

Method of Fractionation.—The distilling apparatus is shown in Fig. 86. The thermometer used is an ordinary thermometer graduated in single degrees, which has been checked against the boiling-point of water. Correction for barometric pressure and exposed stem has not been found necessary, owing to the relatively small amount distilling in the intermediate stages. The diameter of the thermometer, however, should be less than half the diameter of the still-head. The thermometer is placed so that the top of the bulb is level with the side-neck of the distilling flask, and the top of the bead column must be at least ½ in. from the bottom of the bulb. Three 120-c.c. glass-stoppered Erlenmeyer flasks, numbered I, II and III, are used for catching the distillates. Weigh the flasks empty and weigh the sample in flask II.

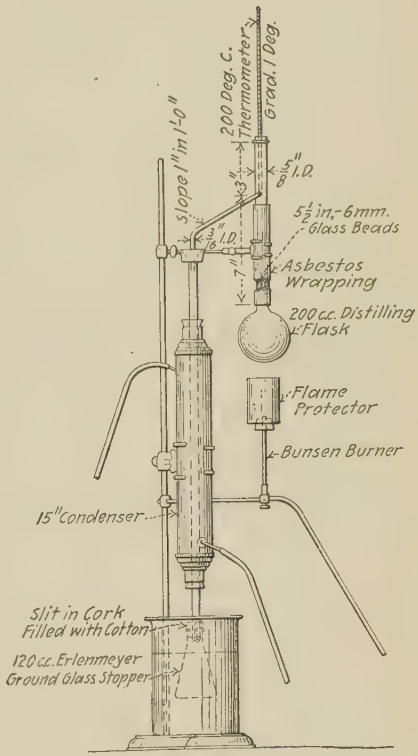

FIG. 86.—Light-oil fractionating still.

Transfer the weighed sample to the distilling flask; distil into flask II with a carefully regulated and protected flame, not faster than one drop per second. At the temperature of 130°C. stop the distillation and allow the condenser to drain well. Replace flask II with flask III, and resume the distillation, stopping when the temperature of 200°C. is reached; then weigh the frac-

tion in flask II (designate as fraction "A") and the fraction in flask III (designate as fraction "B").

Place fraction "A," contained in flask II, in a similar distilling flask and distil to 95°C., catching the distillate in flask I; allow the condenser to drain, replace flask I with flask II, and distil to 120°C. Occasionally the flask will go dry before 120°C. is reached. In such event allow the flask to cool, so that vaporization will not take place when the stopper is removed, and add a small portion of fraction "B" (3 c.c. have been found sufficient in all cases to drive over all the lighter oils), and then resume the distillation. All this process of distillation must be performed slowly, especially when running off the portion between 95° and 120°C.; about 1 drop every 2 seconds is the correct rate.

Now weigh the fraction in flask I (designated as fraction "C") and the fraction in flask II (designated as fraction "D"). Put fraction "C" into a similar distilling flask and re-distil to 95°C., catching the distillate in the same flask, No. I. If difficulty is found in reaching this temperature, owing to the flask going dry, let the flask cool and add a small portion (3 c.c. are sufficient) of fraction "D." This final distillation must be made very slowly, starting with one drop in two seconds and finishing with one drop in four seconds. Finally weigh flask I. Designate its contents as fraction "E."

The weighing flasks are not cleaned between distillations. The distilling flasks and condenser must, however, be clean and dry for each new fractionation.

METHOD OF CALCULATION

Call last fraction to 95° "E."
Call first fraction to 95° "C."
Call fraction 95° to 120° "D."
Call fraction to 130° "A."
Call fraction 130° to 200° "B."

"E" is equivalent to the weight of the true benzene content.

"D" + ("C" − "E") is equivalent to the weight of the true toluene content.

"B" + ("A" − "C" − "D") is equivalent to the weight of the true solvent naphtha content.

In order to figure percentages by volume, take the specific gravity at 15°C. of fraction "E" for the benzene, of fraction "D" for the toluene, and of fraction "B" for the solvent naph-

tha, and make the necessary calculations to percentages by volume of the original light oil. On account of the small size of the distillates, the determination of specific gravity usually will have to be made in each case with a carefully calibrated pycnometer. If possible, use a 10 c.c. pycnometer fitted with a thermometer.[1] These products have large coefficients of expansion, which necessitates careful and rapid weighing.

Correction for Paraffin Hydrocarbons.—In some cases paraffin hydrocarbons may be present and are indicated by low specific gravities of the fractions. The method for calculating the percentage of non-aromatic hydrocarbons is based on Rittman's suggestion.[2] The following figures are for the specific gravities[3] in question:

Specific gravity at 15°C. on	Aromatics	Non-aromatics
Fraction "E".....	0.885	0.720
Fraction "D".....	0.872	0.730
Fraction "B".....	0.869	0.760

The specific gravities of the fractions having been determined, let X = per cent. of aromatics of any fraction of original light oil,

Y = per cent. of non-aromatics of any fraction of original light oil,

G = sp. gr. determined,

A = sp. gr. of aromatic fraction when pure,

N = sp. gr. of non-aromatic constituents, and

P = per cent. of original light oil of fraction under consideration.

$$X = \left(\frac{G - N}{A - N}\right) XP.$$

$$Y = \left(\frac{A - G}{A - N}\right) XP.$$

It is evident that the foregoing method of estimating benzene, toluene, and solvent naphtha is a purely arbitrary one,

[1] Or use the Drushel pycnometer (U. S. Bureau of Mines *Bulletin* **125**, 27), described on page 45.

[2] *Met. Chem. Eng.*, **13** (1915), 682.

[3] Some light oils contain carbon disulphide, the high specific gravity of which may mask completely the presence of any paraffins in the benzol fraction. In such cases the amount of carbon disulphide must be determined (G. LUNGE's "Coal-Tar and Ammonia," 5th ed., 982) and a suitable correction applied.

and it may be open to some criticism on this account; but a considerable amount of experience with it has shown it to give satisfactory results, both by comparison with other methods and by tests on artificial mixtures containing known quantities of the materials in question.

The following table (I) shows the results of estimations on samples made up with known quantities of benzene, toluene and solvent naphtha.

TABLE I

Test	Sample taken, c.c.	Actual composition of mixtures			Estimated by method		
		Benzene, per cent.	Toluene, per cent.	Solvent naphtha, per cent.	Benzene, per cent.	Toluene, per cent.	Solvent naphtha and loss (by diff.), per cent.
1	99.40	50.0	25.0	25.0	49.85	25.12	25.03
2	88.06	67.5	17.5	15.0	66.58	17.63	15.79
3	57.42	70.0	20.0	10.0	69.37	19.55	11.08
4	98.98	75.0	15.0	10.0	74.43	14.71	10.86
5	96.86	80.0	10.0	10.0	80.04	9.25	10.71
6	63.25	45.0	40.0	15.0	41.00	43.69	15.31

Test 6 is illustrative of several experiments that have been made which show that the method is not applicable to mixtures containing less than 50 per cent. of benzene. In cases of such mixtures, if a higher degree of accuracy is necessary, the fractions obtained from the preliminary distillation are mixed together and enough pure benzene is added to bring the composition of the sample within the proper limits.

In Table II are presented the results of distilling samples at a too rapid rate. In test 1 the last fractionation was run at the rate of one drop per second, causing the separation of benzene to be inefficient. Test 2 was run rapidly throughout all the distillations.

TABLE II

Test	Sample taken, c.c.	Actual composition of mixtures			Estimated by method		
		Benzene, per cent.	Toluene, per cent.	Solvent naphtha, per cent.	Benzene, per cent.	Toluene, per cent.	Solvent naphtha and loss (by diff.), per cent.
1	99.40	65.0	20.0	15.0	65.40	18.92	15.68
2	95.83	70.0	15.0	15:0	73.92	13.13	12.95

In Table III are given the results of estimations made by this method compared with analyses of light-oil samples made according to the method of Wilson and Roberts.[1]

TABLE III

Estimated by method given			Test	Estimated by method of Wilson and Roberts		
Per cent. benzene	Per cent. toluene	Per cent. solvent		Per cent. benzene	Per cent. toluene	Per cent. solvent
48.0	20.2	11.0	1	48.9	19.8	12.7
40.0	15.6	....	2	41.4	15.5	6.2
59.1	16.1	11.2	3	60.0	15.3	9.0
44.1	15.9	8.5	4	45.8	15.2	6.2
33.0	30.2	11.7	5	36.0	28.0	11.4

Test 5 shows results obtained from an oil containing too high a percentage of toluene for efficient fractionation by the method given. The benzene content should have been increased by the addition of a known quantity of c. p. benzene, but this was not done in this test.

Approximate Estimation from the Boiling-Point Tests.—It is interesting to note that a quick approximate estimation of the relative proportions of benzene, toluene and solvent naphtha in light oils of ordinary composition may be made directly from the results of the usual "boiling-point tests." The results are of considerable value to the practical benzol-plant operator. The method of making these tests is described on page 218.

The benzene is taken as the average of the amounts distilling at 95° and 100°C.

The toluene is taken as the amount distilled between 100° and 120°C.

Solvent naphtha is taken as $\frac{2}{3}$ of the amount distilling between 120° and 180°C. on unwashed materials, and as the amount distilled between 120° and 180°C. on washed materials.

In the case of light oils containing much over 60 per cent. of benzene, the percentage of toluene as estimated by this method will be considerably too low. When samples contain much over 20 per cent. of toluene, the percentage of toluene as estimated by the method will be considerably too high (see Table V). We have

[1] *J. Gas Ltg.*, May 2, **1916.**

found it to hold fairly good when light oils contain from 45 per cent. to 60 per cent. of benzene, and from 10 per cent. to 20 per cent. of toluene.

Thus suppose we have a light-oil sample which gives the following results by the "boiling-point tests:"

Temperature	Per cent. of total light oil distilling
95°	54
100°	60
120°	78
180°	90

Then the percentage of benzene is the average of 54 and 60 = 57 per cent.

The percentage of toluene is 78 − 60 = 18 per cent.

The percentage of solvent naphtha is 90 — 78 = 12 per cent.

TABLE IV

Results of tests of known mixtures containing correct proportions of benzene, toluene and solvent for estimation by boiling-point method.

Made up from pure materials				Per cent. petroleum wash oil	Estimated by "boiling-points"		
No.	Per cent. benzene	Per cent. toluene	Per cent. solvent		Per cent. benzene	Per cent. toluene	Per cent. solvent
1	60	10	10	20	57.0	9	8
2	55	20	15	10	52.0	17	14
3	50	20	15	15	46.5	17	13
4	45	15	15	25	44.5	14	14

TABLE V

Results of tests of known mixtures containing incorrect proportions of benzene, toluene and solvent for estimation by boiling-point method.

Made up from pure materials				Per cent. petroleum wash oil	Estimated by "boiling-points"		
No.	Per cent. benzene	Per cent. toluene	Per cent. solvent		Per cent. benzene	Per cent. toluene	Per cent. solvent
1	70	15	10	5	71.0	9.0	8
2	30	50	20	..	9.0	66.0	17
3	20	60	20	..	0.5	83.5	16

TABLE VI

Comparison with fractionations, actual coke-oven light oils used.

No.	Fractionated by method of Wilson and Roberts			Estimated by "boiling-points"		
	Per cent. benzene	Per cent. toluene	Per cent. solvent	Per cent. benzene	Per cent. toluene	Per cent. solvent
1	47.5	15.7	8.9	48.0	14	9
2	47.0	14.0	6.0	46.0	14	9
3	46.3	13.8	6.0	48.0	13	9
4	44.8	15.3	7.6	45.5	14	9
5	41.4	15.5	...	40.5	14	10

It will be seen from Table IV that the percentages of light-oil constituents as estimated by the "boiling-point" test run somewhat lower than the true values. However, on an unwashed coke-oven light oil, this test will give values that check the real composition somewhat more closely (*cf.* Table VI). But even then about 1 per cent. should be added to the toluene as determined.

Other methods for the analysis of light oil will be summarized for comparison. We have used all these methods and found them to give good results when proper care is exercised in carrying out the distillations, but they all have their limitations in that they require comparatively large samples.

James[1] distils 250 c.c. of the oil to 140°C. and through a six-bulb LeBel-Henninger column (other efficient columns may be used). The distillate then is distilled, catching six fractions, viz.: to 90°C., 90° to 100°C., 100° to 109°C., 109° to 112°C., 112° to 120°C., and residue at 120°C. All these fractions, except the first and last, are redistilled, using a column as before. All that distils below 110.6° is placed with the first fraction, and all above that with the last. This gives essentially a mixture of benzene and toluene in the first case, and of toluene and xylene in the second. A boiling-point determination of each of the two fractions then is made and the percentage of toluene estimated from tables given. Figures are presented showing the percentage of toluene in artificial mixtures as compared with the percentage estimated by the method. On the whole, very good results were obtained, but this method would not be so applicable to quantities as small as, say, 50 c.c.

[1] *J. Soc. Chem. Ind.*, **35**, 236; *J. Gas Ltg.*, **133**, 531.

Dyke Wilson and Ivan Roberts have described[1] the following method for the determination of benzene, etc.:

Approximately two liters of oil are used. The oil is purified by washing with sulphuric acid, then soda, and the product is distilled with steam. This purified oil is placed in the still. The still is provided with a 40-in. Hempel column filled with glass balls for 36 in. Attached to the top of the Hempel column is an efficient vertical reflux condenser. By adjusting the temperature of the bath of the reflux, any degree of dephlegmation desired may be attained. Benzene is taken as the amount distilling to 81°C. plus benzene in the intermediates. Toluene is taken as the amount distilling between 109° and 111.5°C. plus toluene in the intermediates. Percentages in the intermediates are estimated from boiling determinations in a side-neck flask and comparison with tables. Solvent naphtha is taken as the difference between the amount of washed oil charged and the benzene and toluene as determined, less any distillation losses. Temperatures are measured by an accurate thermometer graduated in fifths of degrees, inserted vertically in the top of the column above the reflux. The advantages of this method are that it gives large volumes of pure materials, only about 4 per cent. of the total washed oil being in the form of intermediates.

Egloff uses a large sample (approximately two liters),[2] which is placed in a copper still of about 4 liters capacity. The column is about 8 ft. in length, made up of six sections of ¾-in. iron pipe, 1 ft. each in length, with a top section 18 in. in length. Sections are connected by means of sleeves. Between adjacent sections there is a 30-mesh iron gauze, used as a diaphragm to support an 8-in. column of 2-in. glass rods of ⅛-in. diameter. Above each section of glass rods a ¼-in. iron pipe is screwed into the column, which, in turn, is screwed into a ¼-in. main running down the outside of the column. This main at the lower end passes into the still, the lower end being just above the bottom, forming a seal when the light oil is charged. This small pipe then acts as a reflux for each of the sections. A thermometer is placed in the top of the column.

Distillation is conducted at a rate of two drops per second. The amount distilling to 95°C. is taken as benzene, the amount

[1] *Gas Record*, March, **1916**; *J. Gas Ltg.*, May 2, **1916**. This procedure sometimes is referred to as the Laclede method.

[2] *Met. Chem. Eng.*, **16**, 259.

from 95° to 125°C. as toluene, and the amount from 125° to 165°C. as the solvent naphtha present in the starting mixtures. Various tables are given which show the efficiency of the fractionation and the accuracy which can be expected.

Egloff purifies the oil before fractionation by washing with sulphuric acid and soda, and then steaming with live steam to 180°C. This oil distillate is dried and placed in the still for fractionation. Paraffins are estimated from the gravity of the fractions obtained.

Rittman[1] has described a method for the determination of benzene in light-oil mixtures by nitration.

METHOD FOR THE DETERMINATION OF WASH OIL IN LIGHT OIL

Take a one liter sample of light oil (noting the temperature at which it is measured) and place in a steam-still of about one gallon capacity. After making sure that all joints and connections are tight, dry-distil to 120°C. Then introduce steam, keeping the temperature between 120° and 130°C. Distil until 400 c.c. of water have passed over. The apparatus is arranged as shown in Fig. 84, except that a one-liter separatory funnel is used. It is well to place an asbestos guard or shield between the separatory funnel and the flame, to minimize the danger of fire. After distillation, the light-oil distillate is brought to the same temperature as the sample as first measured, and its volume is determined by means of a graduate. The volume of the oil left in the still also is measured. This volume added to that of the distillate should equal 1000 c.c. if the still is drained carefully. The discrepancy should not exceed 5 c.c. This residual oil is cooled to 15°C. and the naphthalene is filtered off by suction; after drying as well as possible, the loss in volume of the oil is noted.

A fresh sample of wash-oil of the same type used in scrubbing the gas for light oil is taken for a blank.

Take 1000 c.c. of this for a sample. Distil at 120–130°C. in the same apparatus until 400 c.c. of water have passed over (as in the light oil distillation). Measure the volume of distillate and calculate the amount of residue by difference.

Calculations.

Let A = per cent. of oil residue (corrected for naphthalene) from first distillation.

[1] *Ibid.*, **1915**, 682.

Let B = per cent. of oil residue from blank (second) distillation.
Then: per cent. of wash oil in sample $= \frac{A \times 100}{B}$.

METHOD FOR THE BOILING-POINT TESTS OF BENZOLS

An interesting chapter could be written descriptive of the evolution and standardization of the boiling-point test which is in universal use by all makers and users of the various benzols. It is not, however, our purpose to do this, but we shall give the details of the method that we are employing at present, in the belief that some of these details—which have hitherto been omitted in the descriptions of this method published in various American books and journals—may be found in convenient form to include in standard specifications.

The European method—which is mentioned several times in our description—may be found in Lunge's "Coal-Tar and Ammonia," 4th Am. ed., 755. The American method has been described by S. R. Church (*J. Ind. Eng. Chem.*, **3** (1911), 227). A number of modifications have been adopted more or less widely, such as the substitution of the standard Engler flask for the 200-c.c. Jena flask, and it is probable that the most up-to-date description of the method is that given in the "Gas Chemists' Handbook," recently published by the American Gas Institute (page 180). It is stated that the method in the "Handbook" is adapted with some modification from the report of Sub-Committee D-1, American Society for Testing Materials, 1915. In the notes appended to our description, reference will be made both to the A. G. I. and the A. S. T. M. committee report.[1]

After experience with both the European and the American methods, there can be no hesitation in stating that the latter is superior, but it still has the fault that many of its essential details have not been standardized with sufficient precision.

Flask.—The flask used is the Engler flask, as described on pages 12 and 54. The flask is of globular shape, 6.5 cm. in diameter, with a cylindrical neck, 1.6 cm. in internal diameter and 15 cm. in length. Half way up the side of the neck a vapor tube of not less than 5 mm. internal diameter and not less than 10 cm. nor more than 12 cm. in length extends at an angle of 75° downward to the

[1] See also the standard method for testing paint thinners other than turpentine in the *Appendix*, page 343.

condenser. The junction of the vapor tube with the neck of the flask is 9 cm. above the surface of the oil when the flask contains its charge of 100 c.c. of oil.

NOTE: Redwood's description, as quoted by the A. G. I. and the A. S. T. M., omits the position and diameter of the vapor tube, both of which are important. With a vapor tube too long or too constricted in diameter, a pressure is produced within the flask during distillation, which has a material effect on the boiling-point.

Condenser.—The condenser tube (see Fig. 87) consists of a straight piece of thin glass, 24 in. in length, having an inside

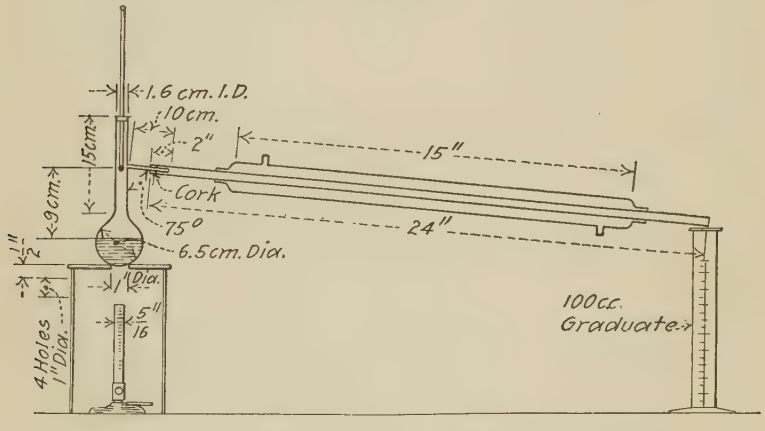

FIG. 87.—Condenser arranged with flask and receiver.

diameter of ½ in. throughout. There are no constrictions or irregularities in the condenser tube. The tube is fitted with a 15-in. jacket having connections for cold water. The tubulature of the distilling flask is inserted through a tightly-fitting cork 2 in. into the condenser tube.

NOTES: The A. G. I. specifies a tube of thin glass, 24 in. in length. It does not specify diameter of tube or length of jacket. The form of condenser is objectionable because the enlarged adapter holds a small amount of benzene and cannot be thoroughly drained in the position shown. The side-neck of the distilling flask should project some little distance beyond the cork, instead of being flush with the cork. The A. S. T. M. condenser is of the trough type, 15 in. long, having a tube of thin brass, ½ in. in internal diameter by 22 in. long. The lower end of the condenser

is cut off at an acute angle and is curved down for a length of 3 in., so as to project at least ½ in. into the 100-c.c. cylinder used as a receiver. A pasteboard cover should be placed over the top of the cylinder and surrounding the condenser tube. The choice between the trough and the ordinary Liebig condenser jacket is simply a matter of personal preference. The same is true of the choice between glass and brass for the condenser tube. In using the trough, arrangements should be made for continuous circulation of water. In benzol work it is not necessary to have the end of the tube curved, as recommended by the A. S. T. M., and the straight tube considerably facilitates cleaning.

Burner.—A standard bunsen burner having separate adjustments for regulating the supply of gas and air is used. The opening at the top of the burner is of 5/16 in. diameter. When making distillations the flame of the burner must be so regulated as to be blue at any portion of the top.

NOTES: A. G. I. and A. S. T. M. give no specifications. The burner recommended is the one most commonly used in this work. The specification regarding the character of the flame is borrowed from European specifications, and is good practice, since a smoky flame may give unsatisfactory results.

Furnace.—The flask rests on an asbestos slab, ⅛ in. thick and 6 in. square, having a circular hole 1 in. in diameter in its center. The furnace is cylindrical in shape, 5 in. in diameter and 8 in. tall. It has four round holes—1 in. in diameter—equally spaced around the top, located ½ in. below the top, for the escape of products of combustion.

NOTES: A. G. I. specifies a 1½-in. opening in the asbestos. A. S. T. M. specifies a 1¼-in. opening. The Barrett Company's laboratories have found a 1-in. opening much more satisfactory, and our own tests confirm this. The following comparisons may be of interest:—

It can be seen readily from these data that the 1-in. opening is the better size, for the following reasons:

1. With a 1-in. opening it is difficult to drive over the first drop too rapidly, and different operators will not vary greatly.
2. It is almost impossible to run the distillation too fast.
3. With only a 1-in. opening the dry-point is obtained much more easily in the case of pure products, for the tendency to superheat is reduced greatly. A larger opening is necessary for higher boiling materials.

Using a 1½-in. opening in the asbestos flame-guard.

	Jena flask	Engler flask
First drop	79.72	79.65
5 per cent	80.12	80.15
10 per cent	80.22	80.25
20 per cent	80.32	70.27
30 per cent	80.32	80.30
40 per cent	80.32	80.33
50 per cent	80.32	80.35
60 per cent	80.32	80.35
70 per cent	80.33	80.35
80 per cent	80.37	80.35
90 per cent	80.42	80.45
Dry	81.02	81.25

Using a 1-in. opening in the asbestos flame-guard.

	Jena flask	Engler flask
First drop	79.87	80.01
5 per cent	80.22	80.33
10 per cent	80.34	80.35
20 per cent	80.36	80.36
30 per cent	80.36	80.36
40 per cent	80.36	80.36
50 per cent	80.36	80.36
60 per cent	80.36	80.36
70 per cent	80.36	80.36
80 per cent	80.37	80.36
90 per cent	80.38	80.37
Dry	80.41	80.44

The shield recommended in the A. G. I. "Handbook" is unsatisfactory because it would be almost impossible to determine the "dry-point" satisfactorily.

The one recommended by A. S. T. M. is rather cumbersome.

The shield recommended above is copied after European practice and has always given very good results.

Receiver.—An ordinary 100-c.c. graduate (graduated in 1 c.c.) is used for the receiver. The graduations must be cut distinctly. The graduate should be approximately 1 in. in diameter. The mark for each 10 c.c. should be longer than the intermediate markings and should be numbered plainly.

NOTE: This is essentially the same as specified by the A. G. I. and the A. S. T. M., but the specifications have been made more definite.

Thermometer.—All distillation thermometers are made of resistance glass of a quality equivalent to suitable grades of Jena or Corning makes, and are annealed thoroughly. They are filled above the mercury with inert gas which will not act chemically on or contaminate the mercury. The pressure of the gas is sufficient to prevent separation of the mercury column at all temperatures of the scale. There is a reservoir above the final graduation large enough so that the pressure will not become excessive at the highest temperatures. The thermometer is finished at the top with a small glass ring or button suitable for attaching a tag. Each thermometer has—for identification—the maker's name and a serial number.

For the boiling determination of crudes and solvent naphthas the thermometer is graduated from 0° to 400°C. at intervals of degrees Centigrade. Every fifth graduation is longer than the intermediate ones, and every tenth graduation beginning at zero is numbered. The graduation marks and numbers conform to the following dimensions:

Total length, mm.	385, maximum.
Diameter of stem, mm.	7; tolerance 0.5.
Diameter of bulb, mm.	5, minimum, and not exceeding that of stem.
Length of bulb, mm.	12.5; tolerance 2.5.
Distance, 0° to bottom of bulb, mm.	30; tolerance 5.
Distance, 0°–400°.	295; tolerance 10.

The accuracy of the thermometer, when delivered to the purchaser, is such that, when tested at full immersion, the maximum error from 0° to 200°C. does not exceed 0.5°; 200°–300°C., 1°C.; and 300°–375°C., 1.5°C. The sensitiveness of the thermometer is such that, when taken at a temperature of 26°C. and plunged into a free flow of steam, the meniscus passes the 90°C. mark in not more than 6 seconds.

For the boiling determination of pure benzene and pure toluene the thermometer is graduated from 70° to 120°C. at intervals of $\frac{1}{10}$°C. Each graduation for half degrees is longer than the intermediate ones, and each degree beginning at 70°C. is numbered. The graduation marks and numbers are clear cut

and distinct. The thermometer conforms to the following dimensions:

Total length, mm	460, maximum.
Diameter of stem, mm	7; tolerance 0.5.
Diameter of bulb, mm	5, minimum, and not exceeding that of stem.
Length of bulb, mm	12.5; tolerance 2.5.
Distance, 70°C. to bottom of bulb, mm	95; tolerance 5.
Distance, 70°C. to 120°C., mm	330; tolerance 10.

The thermometer, when delivered to the purchaser, is furnished with a calibration sheet by the maker, with corrections at 80°C. and 110°C., together with the difference between the expansion of the mercury and glass used, for making the correction for emergent stem. All thermometers are calibrated for total immersion.

NOTES: The thermometer for the determination of crude materials is the same as specified by the A. G. I., and has some slight differences from the A. S. T. M. description. It is necessary, in addition to this, to have a very accurate thermometer for tests of pure benzols. We have found the thermometer described above to be very much more satisfactory than the one described on page 187 of the A. G. I. "Handbook." Most specifications for pure benzene call for a range of 2°. A single interval on the scale of the A. G. I. thermometer amounts to 10 per cent. of this range, and the graduations are so close that it is difficult to in erpolate satisfactorily.

Method of Distillation.—The flask, connected with the condenser, is filled with 100 c.c. of the material at 15.5°C., which is measured in the 100-c.c. receiving cylinder. The same cylinder may be used without drying as the receiving vessel for the distillate. The thermometer is inserted through a tightly fitting cork in the neck of the flask, so that the top of the thermometer button will be on a level with the bottom of the side outlet in the neck of the flask and in the center of the neck. The distillation proceeds at a rate of not less than 4 nor more than 5 c.c. per minute into the receiving cylinder. The flask must be heated very slowly just before the first drop comes over, in order that the thermometer will register the correct temperature. The temperature at which the first drop leaves the end of the condenser is considered the initial boiling point. In testing pure

benzols and refined naphthas, the distillation is continued until the last drop is vaporized, when a puff of white vapor usually appears in the bottom of the flask. The temperature at this point is considered the end- or dry-point of the distillation. The total yield of distillate should not be less than 97 per cent.

NOTES: In making tests of pure benzene, toluene, xylene and refined naphthas, it is our practice to record the following temperature readings: First drop, 5 per cent., 10 per cent., and each 10 per cent. thereafter to the dry-point. The intermediate readings may be omitted at discretion, but often serve a useful purpose in benzol plant operation. In tests of light oils, it is our practice to record the following readings: Temperature of the first drop and per cent. of distillate at the following temperatures: 80°C., 95°C., 100°C., 120°C., 150°C., 180°C., and 200°C. In case the dry-point should come before any of the above temperatures are reached, this is recorded and the distillation discontinued. Doubtful products and residues are distilled in a 200-c.c. glass flask similar to Fig. 85, using a 100-c.c. sample and a thermometer having a range between 70° and 400°C. Temperatures are recorded for the following: 5 c.c., 10 c.c., 20 c.c., 30 c.c., 40 c.c., 50 c.c., 60 c.c., 70 c.c., 80 c.c., 90 c.c., and 95 c.c. In the case of the still residues, the distillation is stopped when the oil becomes extremely thick and viscous. In making boiling-point tests of refined or pure benzols, it is often important that corrections for emergent stem and barometric pressure be made. In the case of crude materials this is not so essential.

Emergent Stem Correction.

Let C = Number of degrees Centigrade to be *added* to the observed temperature.
N = Number of degrees of the stem exposed.
T = Average temperature of the bulb.
t = Average temperature of the stem.
K = Correction factor for the thermometer (*i.e.*, the difference between the coefficients of expansion of mercury and glass).

Then: $C = KN\,(T - t)$.

For example:

Observed temperature	= 80°C.
Steam temperature	= 25°C.
Degrees emerging (from top flask to 80°C.)	= 15°C.

Thermometer coefficient	= 0.000154.
80°C. − 25°C.	= 55°C.
15° × 55° × 0.000154	= 0.12787 = 0.13°C.
Temperature corrected for emergent stem	= 80.13°C.

Barometric Pressure Correction.—The difference of temperature for each millimeter between 720 mm. and 780 mm. is in the case of:

Benzene	0.043
Toluene	0.047
Xylene	0.052
50 per cent. benzol	0.0461
90 per cent. benzol	0.0453

For example: If the barometer reading is 754.7 mm. (at 0°C.) and the thermometer (corrected for emergent stem) is 80.13 in the case of benzene:

760 − 754.7 = 5.3 mm.

0.043 × 5.3 = 0.2279 = 0.23°C. to be added.

80.13° + 0.23° = 80.36°C. corrected temperature.

NOTE: It must be borne in mind that, if the barometer reads above 760 mm., the barometric correction must be subtracted.

METHOD FOR DETERMINING THE SPECIFIC GRAVITIES OF BENZOLS

For routine purposes the test is made by means of a Westphal balance.

Cool the oil to about 12°C., rinse out the cylinder with a portion of the oil; suspend the plummet from the balance into the cylinder, and fill the latter with the cold oil. When the temperature, as shown by the plummet thermometer, is almost 15.5° C., place weights on the beam and adjust rapidly at 15.5°C. The specific gravity may be determined by a suitable pycnometer, but extra precautions have to be taken on account of the high coefficients of expansion of the benzols.

SULPHURIC ACID COLOR TEST FOR BENZOL, TOLUOL, SOLVENT NAPHTHA, ETC.

The set of color standards consists of fifteen sealed tubes approximately 1 in. in diameter, each containing one of the solutions made up as given below.

When making a test for amount of acid washing, a glass-stoppered weighing bottle, about 1 in. in diameter and 2½ in. high, is used (E. H. Sargent No. 802[1]). Sufficient concentrated sulphuric acid is put in to fill ¼ of the bottle, and then 3 times as much of the sample to be tested is added. Shake thoroughly for 15 to 20 sec., and allow to stand for specified time. Compare the resulting color of the acid layer with the standard set, and determine to which number it corresponds.

In pure benzene and pure toluene testing, the benzene or toluene layer must remain white, and the color of the acid layer, after standing 15 min., must not be darker than No. 4.

For 90 per cent. benzol and all grades of benzol and toluol other than pure, the benzol and toluol layer must remain white and the color of the acid layer, after standing 15 min., must not be darker than No. 6.

For solvent naphtha the acid layer color only is noted. After 5 min. standing it must not be darker than No. 14.

In making the test it is customary to take the color of the main portion of the acid, and should a darker ring form—just below the benzol or toluol—this is disregarded.

The following basic solutions are used in making the standards:

(A) 59.4965 g. $CoCl_2.6H_2O$ (nickel-free) is made up to 1000 c.c. with a mixture of 25 c.c. 31 per cent. HCl and 975 c.c. H_2O.

(B) 45.054 g. $FeCl_3.6H_2O$ made up to 1000 c.c. with a mixture of 25 c.c. 31 per cent. HCl and 975 c.c. H_2O.

(C) 3.5 volumes of solution A + 36.5 volumes solution B + 90 volumes of H_2O.

(D) 3.5 volumes of solution A + 36.5 volumes of solution B (no water is added).

(E) Solution of K_2CrO_4, saturated at 21°C.

(F) 1 volume of a solution of $K_2Cr_2O_7$ saturated at 21°C. + 1 volume of H_2O.

As standard color solutions to be used for comparison the following are made up and numbered from 0 to 14:

No. 0. Pure water.
No. 1. 1 volume of solution C + 1 volume of H_2O.
No. 2. 5½ volumes of solution C + 2 volumes of H_2O.
No. 3. Solution C as such.

[1] One-ounce glass-stoppered "French Squares" usually are specified. We have found that the round bottles with straight sides are easier to clean.

No. 4. 1 volume of solution D + 1 volume of H_2O.
No. 5. 5½ volumes of solution D + 2 volumes of H_2O.
No. 6. Solution D as such.
No. 7. 5 volumes of solution E + 2 volumes of H_2O.
No. 8. Solution E as such.
No. 9. 7 volumes of solution E + ½ volume of solution F.
No. 10. 6½ volumes of solution E + 1 volume of solution F.
No. 11. 5½ volumes of solution E + 2 volumes of solution F.
No. 12. 1 volume of solution E + 1 volume of solution F.
No. 13. 2 volumes of solution E + 5 volumes of solution F.
No. 14. Solution F as such.

TESTS OF WASHED BENZOL

These tests are made on samples of benzol from the agitator which is used for the sulphuric acid treatment of the crude distillates. The object is to determine whether a sufficient degree of purification has been attained by the acid treatment.

Wash Test for Benzol and Toluol.—It is presumed that the benzol or toluol or mixture of both has been agitated with sulphuric acid and the latter removed after the customary settling. Transfer about 350 c.c. of the sample into a 500-c.c. separatory funnel; add about 30 c.c. of tap water; shake about 30 sec., and allow to settle. Drain off water and impurities. Add about 30 c.c. of a 1.10 sp. gr. solution of sodium hydroxide (95 g. per liter), and agitate about 50 sec. or until the mixture develops a yellowish color. Let settle and drain off the soda and impurities. Add about 30 c.c. of tap water; agitate about 30 sec.; let settle and drain off water, leaving the completely washed benzol and toluol in the funnel. Add between 5 and 10 g. of fused calcium chloride, agitate for a few seconds, and allow to settle until the oil clears. Transfer through the mouth of the funnel about 60 c.c. of the oil into a clean 100-c.c. distillation flask, previously rinsing the flask with the same oil, exercising care to prevent the benzol from getting into the side neck of the flask. Connect to a clean condenser and distil to about 80°C., catching the distillate in a clean test-tube. Quickly reject this distillate from the test-tube and continue the distillation to 95°C. in the case of benzol, or to 115°C. in the case of toluol or a mixture of benzol and toluol. The sulphuric acid color test then is made on a portion of the distillate, as described in the preceding test. Care must be taken to test each lot of calcium chloride for impurities that might affect the results.

Wash Test for Solvent Naphtha.—It is presumed that the solvent naphtha has been agitated with sulphuric acid; then allowed to settle, and separated. Transfer about 350 c.c. to a 500-c.c. separatory funnel and add about 25 g. of calcium oxide (pulverized). Shake about 1 min. and allow to settle. Rinse a clean 100-c.c. distilling flask with a portion of the solvent and distil 60 c.c. to 180°C., using a clean condenser and test-tube for the distillate. The sulphuric acid color test then is made on a portion of the distillate, as described above.

TEST FOR SULPHUR DIOXIDE AND SULPHUR TRIOXIDE IN WASHED BENZOLS

Place the oil to be tested in a bottle, filling the bottle not more than half. Cork and shake well; then uncork and quickly place a piece of moist test paper in the mouth of the bottle, holding by the fingers or by carefully replacing the cork. The paper must in any case be exposed to the vapors above the oil. The test is quite sensitive and the color change will be noted almost immediately if SO_2 or SO_3 is present. The test paper will change from a yellow to a red color in the presence of SO_2 or SO_3.

Alternate Method.—This test can be made very conveniently in connection with the test for completeness of washing with acid. Before distillation of the washed light oil is made, place an adapter on the end of the condenser, and insert a piece of the moist test paper in the top of the adapter in such a manner that the paper is exposed to the vapors, but not to the stream of condensed liquid. The moist paper will adhere quite readily to the walls of the adapter. Continue with distillation and note the color change if SO_2 or SO_3 is present.

To Make Test Papers.—Make a solution of methyl orange by placing 0.1 g. of it in 100 c.c. of water, shaking it at intervals and finally allowing to stand over night. Finally filter off any solid matter and use the clear liquid for making test papers. Cut up the filter paper into narrow strips (paper free from sulphites and acids should be used) and dip into the methyl orange solution. Shake off the excess solution, as the test gives best results with moist paper, *i.e.*, paper that has no drops of excess solution adhering to it.

APPROXIMATE DETERMINATION OF UNSATURATED COMPOUNDS IN WASHED BENZOL BY BROMINE TITRATION[1]

A measure of the quantity of unsaturated compounds (hydrocarbons of the ethylene and acetylene series, etc.) present in crude benzol is afforded by titration with bromine. The bromine water formerly used for this purpose is replaced best by a solution of potassium bromide and bromate, which, on addition of sulphuric acid, sets free 8.0 g. of bromine per liter. The test is carried out by Frank in the following manner:

5 c.c. of the sample are added to 10 c.c. of dilute sulphuric acid (1:5) contained in a 50-c.c. bottle provided with a glass stopper, and N/10 bromide-bromate solution containing 9.9167 g. KBr + 2.7833 g. $KBrO_3$ per liter is added from a burette until, after 5 minutes' shaking, bromine still remains. The titration is finished when the floating oil remains orange-red in color after standing for 15 minutes, and when one drop gives a dark blue coloration with freshly prepared potassium iodide-starch paper. The quantity of bromine used (1 c.c. = 0.008 g. Br) should be reported as grams of Br per 100 c.c. of sample. To obtain accurate results, a preliminary determination is made first, and then two exact estimations, the mean of which is taken. Pure benzene and pure toluene should show a marked bromine reaction after the addition of 0.1 c.c. of the bromine solution; while 90 and 50 per cent. benzols will decolorize on an average 0.6 c.c. of the reagent, but seldom more than 1 c.c. Commercial xylene absorbs 2 per cent. of bromine in a few minutes and considerably more on standing for some time.

FREEZING-POINT TEST OF BENZENE

The thermometer used is a so-called "titer-test" thermometer, graduated to tenths of a degree from 0° to 65°C., and is about 15 in. long. Those in use at present are made by the Taylor Instrument Companies (Catalog No. 1425). They should be standardized against the freezing-point of water. About 40 c.c. of

[1]Method of Kraemer and Spilker (Muspratt's "Encyclopaedia" (**1905** German ed.), **8**, 332 *et seq.*). On the estimation of paraffins and unsaturated compounds in commercial toluols, see Evans, *J. Soc. Chem. Ind.*, **38** (1919), 402T; Lewis, *ibid.*, **39** (1920), 50T; and Colman, *ibid.*

pure benzene are placed in a tube or short cylinder—preferably a titer cylinder, of about 1¼-in. diameter and about 3½ in. high, made of rather thin glass. This is placed in a bath of ice water, and a small amount of salt is added. The bath temperature should be about 0°C.

Suspend the thermometer in the benzene, stirring constantly. When the temperature remains constant about 30 seconds (care must be exercised here not to supercool the benzene greatly; however, with stirring, there is not so much danger of this), allow the thermometer to stand with the bulb in the center of the benzene, and allow the mercury to rise. The highest point is the freezing-point.

The following comparisons will show the relative sensitiveness of the freezing-point and boiling-point tests. First, samples of pure benzene were subjected to the boiling-point and freezing-point tests, with the following results:

	1 Deg. C.	2 Deg. C.	3 Deg. C.	4 Deg. C.
First drop	80.15	79.63	79.68	78.72
5 c.c	80.25	80.13	80.06	79.45
10 c.c	80.32	80.21	80.16	79.67
20 c.c	80.35	80.28	80.24	79.92
30 c.c	80.35	80.35	80.31	80.09
40 c.c	80.36	80.39	80.36	80.22
50 c.c	80.36	80.42	80.36	80.27
60 c.c	80.37	80.43	80.36	80.32
70 c.c	80.37	80.47	80.36	80.37
80 c.c	80.38	80.53	80.41	80.42
90 c.c	80.42	80.58	80.46	80.47
95 c.c	80.45	80.68	80.53	80.52
Dry	80.47	80.93	80.61	80.72
Freezing-point	5.43	5.15	5.15	4.70

To show the relative effects of small amounts of impurities, comparisons were made on samples of pure benzene to which 0.5 per cent. of toluene, petroleum ether and carbon disulphide had been added, with the following results:

Portions of sample No. 1 (above) were used.

	Benzene, 99.5 per cent. Toluene, 0.5 per cent. Deg. C.	Benzene, 99.5 per cent. Petroleum ether, 0.5 per cent. Deg. C.	Benzene, 99.5 per cent. Carbon disulphide, 0.5 per cent. Deg. C.
First drop	80.23	79.59	79.18
5 c.c.	80.53	80.11	79.68
10 c.c.	80.54	80.30	79.88
20 c.c.	80.58	80.48	80.08
30 c.c.	80.64	80.49	80.18
40 c.c.	80.66	80.49	80.26
50 c.c.	80.68	80.57	80.34
60 c.c.	80.72	80.58	80.41
70 c.c.	80.74	80.59	80.43
80 c.c.	80.77	80.62	80.45
90 c.c.	80.94	80.62	80.48
95 c.c.	81.83	80.62	80.48
Dry	83.35	80.63	80.53
Freezing-point	5.2	5.3	5.0

EVAPORATION TEST OF BENZOLS[1]

2 c.c. of the benzol are placed on a metal surface with raised edges 3.5 in. square, and the time taken for complete evaporation is noted. In order to allow for variation in temperature, this should be compared with pure benzene, which requires 10 minutes for complete evaporation at ordinary laboratory temperatures.

METHOD FOR THE DETERMINATION OF CARBON DISULPHIDE IN BENZOLS[2]

Fifty g. of the benzol are mixed with 50 g. of a solution of potassium hydroxide and absolute ethyl alcohol (11 g. of potassium hydroxide and 90 g. of absolute alcohol). The mixture is shaken and allowed to stand several hours at room temperature. About 100 c.c. of water then are added. The aqueous solution is separated from the benzol and the latter is washed several times with water. The washings are added to the aqueous solution. The potassium thiocarbonate is determined either in the solution directly or in an aliquot part thereof, by means of a solution containing 12.475 g. of crystallized

[1] *J. Ind. Eng. Chem.*, **3**, 11.

[2] A. Spilker's "Kokeri und Teerprodukte der Steinkohle."

copper sulphate per liter. One cubic centimeter of this solution is equivalent to 0.0076 g. of carbon disulphide. The aqueous solution is neutralized with acetic acid and the standard copper solution is added until a drop of the solution put on a piece of filter paper, by means of a glass rod, shows a red color when brought in contact with a drop of potassium ferrocyanide. The approach of the end-point is made known by a certain agglomeration of the finely divided precipitate of copper thiocarbonate.

The amount of potassium hydroxide solution given above is sufficient for a benzol containing 5 per cent. of carbon disulphide, but for larger percentages of carbon disulphide the quantity of potassium hydroxide must be increased, or else a smaller sample of benzol must be taken.

It is important to run a careful blank on the reagents used.

DETERMINATION OF HYDROGEN SULPHIDE IN LIGHT OIL

Place 50 c.c. of the light oil in a separatory funnel, and shake thoroughly with a mixture of 20 c.c. of normal sodium hydroxide solution and 50 c.c. of water. After shaking, let the oil separate and draw off the aqueous layer. Wash the light oil again by shaking two or three times with water, separating the aqueous layer each time. Unite the aqueous extracts; dilute to 300 or 400 c.c.; add dilute HCl in excess, and titrate with N/10 iodine solution, using starch as an indicator. One c.c. of N/10 iodine is equivalent to 0.0017 g. of H_2S.

SPECIFICATIONS AND TESTS OF WASH-OIL USED FOR BENZOL RECOVERY

1. Specific gravity at 15°C. must not be over 0.880.

2. The viscosity must not be more than 92 seconds at 21°C., and must not be more than 150 seconds at 4°C., both tests to be made in a Saybolt viscosimeter. No solid matter must separate at 4°C.

3. At least 95 per cent. of pure oil should separate from the water in 10 minutes and the oil should not contain more than 0.5 per cent. of water in the separated oil when the following test is made.

Twenty c.c. of distilled water and then 100 c.c. of the oil are placed in a graduated 250-c.c. cylinder. Steam is bubbled

through the two liquids by means of a glass tube, whose inside diameter is approximately $\frac{3}{16}$ in. After steaming 10 minutes, the cylinder and its contents are placed in a water-bath at 15°C. for 10 minutes, and the volume of separated oil is read. To determine the amount of pure oil separated and water content of the separated oil, draw off the oil by means of a pipette, taking care to get none of the water layer; shake the oil thoroughly; take 20 c.c. and mix with 80 c.c. of pure solvent naphtha in a graduated precipitating tube having the lower end drawn out and graduated in $\frac{1}{10}$ of 1 c.c. This oil-solvent naphtha mixture is kept at a temperature of not over 27°C. for one hour, or the mixture may be separated by means of a centrifuge. The amount of water separated then is read.

4. The oil must not contain more than 12 per cent. of olefines, as determined by the following test:

The apparatus consists of a glass-stoppered flask having a 100-c.c. mark at the bottom of the neck and a 110-c.c. mark at the top of the neck. (This is listed as a "Cassia flask" by most dealers in chemical apparatus.) The space between these two marks is divided into $\frac{1}{5}$ c.c. or $\frac{1}{10}$ c.c. Special sulphuric acid is prepared by mixing one part of fuming sulphuric acid (containing 25 per cent. of SO_3) with two parts of concentrated sulphuric acid containing 93 per cent. of H_2SO_4.

Ten c.c. of the wash oil are measured exactly by a burette into the 100-c.c. graduated flask and shaken vigorously with 25 c.c. of the special acid. After standing for one hour, fill the flask to the 100-c.c. mark with concentrated sulphuric acid. The acid should be added by pouring it slowly against the neck of the flask in order to avoid agitating the already settled mixture. Allow the mixture to stand for two hours and then read the amount of the paraffins left. The difference gives the amount of olefines in the original 10 c.c. of oil. This test is to be carried out at a temperature of 25°C.

5. Not more than 5 per cent. of the oil should distil under 250°C., and an additional 90 per cent. should distil within 120°C.

Notes on Specification Tests.—*Specific Gravity*: The determination is made with a Westphal balance. In setting up the balance, the screw in the base of the balance is adjusted so that the two-pointer protuberances are exactly on the same level when the thermometer is suspended at the end of the beam. The height of the beam can be regulated by the screw in the vertical

support so that the plummet does not touch the bottom of the glass cylinder. The weights consist of four sizes. The largest size hung on the hook at the end of the beam gives units and on any other space it gives tenths; the next size indicates hundredths; the next thousandths, and the smallest ten-thousandths of specific gravity.

After cooling a sample of the oil to about 10°C., the cylinder is rinsed therewith, and the suspended plummet is inserted in the cylinder, which is filled with the cooled oil. The weights are placed on the various divisions and so adjusted that when the thermometer indicates 15.5°C., the two-pointer protuberances are on the same level. (After the specific gravity figure is obtained, the plummet and cylinder are rinsed with benzol.)

Distillation: The distillation is made in the apparatus described in Fig. 88, using 200 c.c. of the oil as a sample. The temperature is recorded for the following quantities of distillate:

First Drop

Cubic centimeters	Per cent.	Cubic centimeters	Per cent.
10	5	100	50
20	10	120	60
40	20	140	70
60	30	160	80
80	40	180	90

Olefines: The special acid, equivalent to two parts of 93 per cent. H_2SO_4 and one part of fuming sulphuric acid containing 25 per cent. of free SO_3 by volume, may be prepared from acids of these strengths by the following method:

According to Van Nostrand's "Chemical Annual," 93 per cent. H_2SO_4 contains 1393 g. of SO_3 per liter. The 25 per cent. fuming sulphuric acid contains 86.22 per cent. of total SO_3. Calculation shows that the acid required contains 1485 g. of SO_3 per liter. An example will illustrate the calculations necessary to prepare this acid.

To make up 1000 c.c. of H_2SO_4 containing 1485 g. per liter of SO_3 from 95 per cent. (conc.) H_2SO_4 and fuming H_2SO_4 (15 per cent. free SO_3).

Then: Let X = number of c.c. of 95 per cent. acid required.
$1000 - X$ = number of c.c. of 15 per cent. free SO_3 acid required.

Total SO_3 in X c.c. of 95 per cent. H_2SO_4 = 1.426Xg.
15 per cent. fuming acid contains 84.39 per cent. of total SO_3.
Specific gravity of 15 per cent. fuming acid is 1.918.
SO_3 in (1000 − X) c.c. of 15 per cent. fuming H_2SO_4 = 0.8439 × 1.918(1000 − X) g.

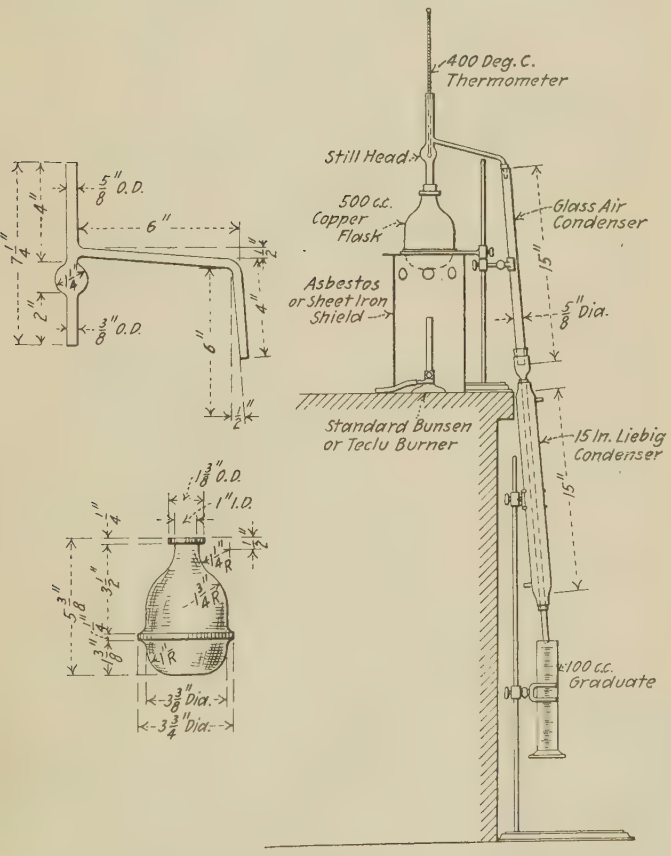

FIG. 88.—Apparatus for the distillation of straw oil.

1.426X + 0.8439 × 1.918 (1000 − X) = 1485.
X = 696 c.c. of 95 per cent. (conc.) H_2SO_4.
1000 − X = 304 c.c. of 15 per cent. free SO_3 fuming H_2SO_4.

THE DETERMINATION OF LIGHT OIL IN BENZOLIZED AND DEBENZOLIZED WASH OIL

Benzolized Wash Oil.—One liter of the benzolized oil is transferred to a ½-gallon copper still. The still is connected to a 15-in. condenser leading into a 500-c.c. separatory funnel, described in the method for "Determination of Light Oil in Gas" (Fig. 84). The oil is heated to about 150°C. with a burner, and live steam then is conducted through the oil at such a rate that the distillate comes over quite rapidly, but not in a continuous stream. Unusual care should be taken to attach the cover to the top of the still in such a way that the latter does not leak. The presence of a leak, no matter how slight, will produce false results. The lid, provided with a firm cardboard gasket, should be fastened on as tightly as possible, and the space between the edge of the lid and the thread of the still should be wrapped tightly with asbestos cord. This wrapping of asbestos cord can be made absolutely vapor-tight by cementing with sodium silicate; but this is not always necessary.

The oil should be submitted to the steam distillation until 500 c.c. of water are condensed in the funnel. The distillate of oil in the funnel then is drained free of water and is agitated with about 10 c.c. of a saturated solution of calcium chloride, which finally is drawn off. The inside of the stem of the funnel is wiped thoroughly dry by means of a pellet of raw cotton and the oil distillate is transferred to a 100-c.c. flask (Fig. 85), in which it is dry-distilled to 200°C., the distillate being received in a graduated test-tube.

$$\frac{\text{No. c.c. of distillate up to 200° C.} \times 100}{1000} = \text{per cent. of light oil in the benzolized absorbent oil.}$$

Debenzolized Wash Oil.—The procedure is similar to the method used in the case of benzolized wash oil, with the exception that a sample of two to four liters is used. This is distilled in a similar still, and the distillation is considered complete when two liters of water have condensed in the separatory funnel.

DETERMINATION OF NAPHTHALENE IN OILS[1]

The apparatus is shown in Fig. 89.

The sample, about 10 g. (the quantity depending upon the

[1] Adapted from the method for naphthalene in gas, "Gas Chemists' Handbook," 1916.

approximate naphthalene content[1]), is weighed accurately and placed in the test-tube, which is an 8-in. by 1-in. flat-bottom test-tube. The 6-in. calcium chloride tube (empty) serves as a guard; it is connected as shown by glass tubing through a two-holed rubber stopper, practically to the bottom of the flat-bottom test-tube. The guard tube serves as a safety device in case the oil is blown out. It also serves as the air inlet for the air which passes through the oil and carries out the naphthalene. The water-jacketed condenser has an 18-in. water jacket and an inner

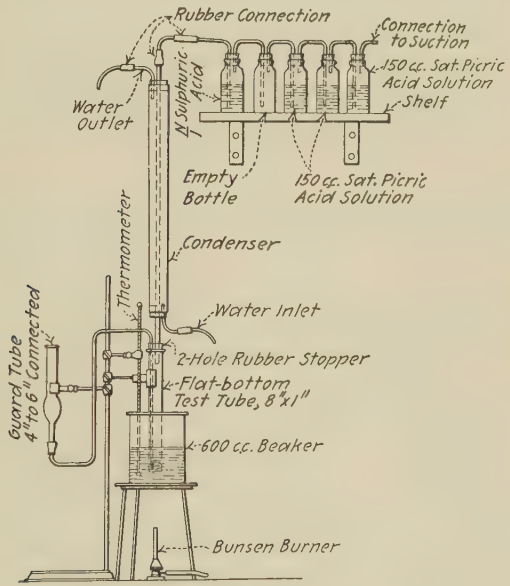

FIG. 89.—Apparatus for the determination of naphthalene in oils.

tube of 12 mm. This conveys over the air with the naphthalene, but returns the oil to the test-tube. The first wide-mouth salt bottle contains about 200 c.c. of N/1 sulphuric acid. This bottle will trap any tarry or alkaline substances. Next is an empty bottle serving as a trap for any solvent carried over.

[1] Enough naphthalene should be present to saturate the picric acid in the first absorption bottle. This is important for obtaining accurate results. When too little naphthalene is present the results will tend to be too high. In such cases we have found it desirable to add a measured volume of benzol containing a known amount of naphthalene to the solution in the 250 c.c. flask before making up to the mark. This is corrected for in the final result.

The sample is placed in the test-tube, and the apparatus is connected and the suction turned on so that air bubbles through the oil at about 1½ cu. ft. per hour. Water is turned on in the condenser. The burner is placed under the beaker containing the water; the vessel is heated slowly until the water boils, and this temperature is continued for one hour. Then the flame is turned out and the air is allowed to bubble over night.

Preparation of the Benzol Solution of Naphthalene Picrate.—Transfer the picric acid solution and the precipitate to a liter separatory funnel, the residues being washed in with naphthalene-free benzol. Shake the contents of the funnel gently until the precipitate is dissolved completely in the benzol. Reject the aqueous layer and draw the benzol solution into a 250-c.c. measuring flask. Make up to the mark with benzol and thoroughly mix.

Determination of the Total Picric Acid.—Titrate 50 c.c. of this solution—preferably in a 200–250-c.c. separatory funnel, for shaking is necessary—with N/5 sodium hydroxide solution, using methyl red as the indicator. This titrates both the free and the combined picric acid.

If T = c.c. of N/5 NaOH required for this total titre, then $5T$ = c.c. of N/5 NaOH required for the total titre of the whole solution.

(To prepare methyl red indicator, dissolve 2 g. of methyl red in 1 liter of a mixture of two parts of 95 per cent. ethyl alcohol and one part of water.)

Determination of the Free Acid.—The free picric acid present is determined by the following procedure:

From a burette draw $\frac{1200}{T}$ c.c. of the benzol solution into a 100-c.c. flask and evaporate to dryness to remove the benzol, proceeding carefully according to the following directions. The flask is placed in a hot water-bath, and a current of air is passed over (not in) the benzol solution, at no time allowing the level of the hot water to be above the level of the benzol solution. The flask should be shaken gently during the evaporation to keep the walls moist and to avoid over-heating any part. Keep the flask in the hot water about one minute after the residue appears dry, then remove, but continue the air current until the odor of benzol no longer can be detected.

Dissolve the residue in the flask with 10 c.c. of 95 per cent. ethyl alcohol, heating gently, if necessary; then precipitate the naphthalene picrate with distilled water, adding it slowly with agitation until the volume is 100 c.c. The temperature of this solution must be cooled to 20°C.—in no case more than 2° higher or lower.

Filter through a dry filter into a 100-c.c. cylinder and titrate 90 c.c. of the filtrate with N/5 sodium hydroxide solution, using methyl red as the indicator.

The number of c.c. of N/5 NaOH taken divided by 0.9 gives the titre required for the free acid (picric) in $\frac{1200}{T}$ c.c. of the original benzol solution.

If F = c.c. of N/5 NaOH required for this $\frac{1200}{T}$ c.c.,

then $F \times \frac{250}{\frac{1200}{T}}$ = NaOH required for the free picric acid in the whole benzol solution.

Calculation for the Naphthalene from the Picric Acid Titres.—The difference between the total titre and the free acid titre is the titre of the picric acid combined as naphthalene picrate, and this latter titre multiplied by its naphthalene equivalent gives the grams of naphthalene in the sample.

$$\left\{5T - F \times \frac{250}{\frac{1200}{T}}\right\} \times 0.0256 = \text{g. of naphthalene in the sample,}$$

or, simplified:

$0.0053T\ (24 - F) =$ g. of naphthalene in sample; and

$$\frac{0.0053T\ (24 - F)}{\text{grams of sample}} \times 100 = \text{percentage of naphthalene.}$$

Notes on the Method.—The reactions involved in the method are the following. Naphthalene combines with picric acid to form naphthalene picrate, according to the following equation:

$$C_{10}H_8 + C_6H_2(NO_2)_3OH = C_6H_2(NO_2)_3OHC_{10}H_8 + H_2O;$$

$$\underset{\text{Picric Acid}}{C_6H_2(NO_2)_3OH} + NaOH = \underset{\text{Sodium Picrate}}{C_6H_2(NO_2)_3ONa} + H_2O;$$

$$C_6H_2(NO_2)_3OH.C_{10}H_8 + NaOH = C_6H_2(NO_2)_3ONa + C_{10}H_8 + H_2O.$$

To absorb the naphthalene completely, the picric acid solution must be saturated fully, and to insure this an excess of crys-

tals must be present. The reason for taking $\frac{1200}{T}$ of the benzol solution for the free picric acid test is that this is the quantity which contains the right amount of picric acid (1.1 g.) to saturate the 100 c.c. of solution to which it is finally made up, and this saturation is essential to prevent decomposition of the naphthalene picrate present at the stated temperature (20°C.). Naphthalene picrate is decomposed easily by heat, evolving naphthalene and leaving behind free picric acid. A blank should be run on the reagents, using a weighed amount (about 0.1 g.) of pure naphthalene dissolved in 250 c.c. of benzol.

PROPERTIES OF THE IMPORTANT CONSTITUENTS OF LIGHT OIL

Properties	Data				
	Carbon disulphide, CS_2	Benzene, C_6H_6	Toluene, C_7H_8	m-Xylene, C_8H_{10}	Naphthalene, $C_{10}H_8$
Molecular weight (0 = 16)	76.12	78.05	92.06	106.08	128.06
Lb. per U. S. Gal. (60°F.)	10.57	7.36	7.27	7.26	9.60
Sp. gr. (0°C./4°C.)	1.2921	0.8999	0.8845	0.8823	
Sp. gr. (10°C./4°C.)	1.2773	0.8893	0.8757	0.8738	
Sp. gr. (15°C./4°C.)	1.2698	0.8839	0.8714	0.8697	1.1517
Sp. gr. (20°C./4°C.)	1.2623	0.8786	0.8659	0.8655	
Sp. gr. (30°C./4°C.)	1.2473	0.8679	0.8573	0.8574	
Expansion coefficient (°C.)	0.00125	0.0012	0.0010	0.00095	
Boiling-point (760 mm. Hg.)	46.2	80.36	110.3	139.1	217.7
Increase in boiling-point (°/mm. Hg.)	0.041	0.043	0.047	0.052	0.059
Vapor pressure mm. Hg. (0°C.)	127.9	26.63	7.20	1.75	0.022
Vapor pressure mm. Hg. (10°C.)	198.5	45.68	13.02	3.45	0.047
Vapor pressure mm. Hg. (15°C.)	244.1	58.90	17.22	4.74	0.062
Vapor pressure mm. Hg. (20°C.)	298.0	75.21	22.53	6.43	0.080
Vapor pressure mm. Hg. (30°C.)	434.6	119.34	37.46	11.43	0.135
Lbs per cu. ft. vapor (60°F.–30″)	0.202	0.209	0.244	0.281	0.339
Kg. per cu. m. vapor (0°C.-760 mm.)	3.42	3.54	4.14	4.76	5.72
Heat combustion (net), 15°C.–760 mm. Hg.:					
Cal. per kg. liquid	3,480	9,960	10,150	10,230	9,700
Cal. per liter liquid	4,420	8,805	8,850	8,910	11,170
B.t.u. per lb. liquid	6,260	17,930	18,270	18,410	17,460
B.t.u. per U. S. gal. liquid	66,100	132,100	132,600	133,500	167,300
Cal. per cu. m. vapor	11,550	33,600	40,150	46,500	52,400
B.t.u. per cu. ft. vapor	1,300	3,780	4,500	5,210	5,910
Specific heat (gal. per kg.)	0.240	0.419	0.440	0.383	0.314
Heat vaporization (cal. per kg.)	83.8	92.9	83.55	78.25	
Solubility in water (22°C.):					
G. substance in 100 g. H_2O	0.219	0.072	Insol.	Insol.	Insol.
G. H_2O in 100 g. substance	0.765	0.241	Insol.	Insol.	Insol.
Melting-point (°C.)	−108.6	+5.4	−92.4	−54.8	+80.0

CHAPTER X

THE EXAMINATION OF NATURAL GAS

1. Sampling.
2. Density:
   (*a*) Effusion Method.
   (*b*) Gas Density Balance.
3. Heating Value.
4. Analysis of Natural Gas.
5. Gasoline in Natural Gas:
   (*a*) Bureau of Mines Method.
   (*b*) Charcoal Absorption Method.
   (*c*) Other Methods.
6. Evaluation of Natural Gas for Carbon Black.
7. The Measurement of Natural Gas:
   (*a*) Calibration of Meters.
   (*b*) Testing Natural Gas Wells for Volume.

1. Sampling.—In collecting samples of natural gas in the field, containers such as those shown in Fig. 90 generally are used. The two valves are opened and the gas is passed through the vessel until it is certain that all the air is displaced, before closing the valves or cocks. Frequently the sampling is carried out by first filling the container entirely with water, attaching one of the nipples to the source of supply, and opening the upper valve and then the lower, thus allowing the water to flow out and the gas to enter. Finally, the lower valve is closed and then the upper, the sample being secured in this way under slight pressure.

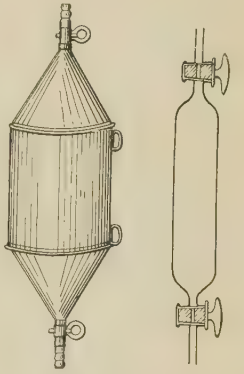

FIG. 90.—Gas sampling tubes.

An ordinary bottle[1] may be used as a sample container. In this case the sample of gas usually is collected as follows: The bottle is

[1] The 16-oz. magnesium citrate bottle which is provided with a rubber gasketed patented stopper is used very widely in procuring gas samples. A bottle of this description can be purchased at any pharmacy. Air displacement is considered by some experts as a trustworthy means of obtaining a sample and unless the gas is not to be analyzed immediately it is unnecessary to paraffin the container.

filled entirely with water and inverted in a tank of water. The gas then is allowed to enter and displace the water in the bottle. With the neck of the bottle still submerged, the cork is inserted tightly, the bottle removed and the cork wired on or tied securely with strong string. Finally, the cork is covered with paraffin. For collecting larger samples, often under pressure, a steel cylinder similar to an oxygen tank generally is used. Care should be exercised in this operation to ensure that the air entirely is displaced before closing the valve or applying the pressure.

When securing a sample from a smaller line connected to a main in which the pressure is greater than atmospheric, the line should be purged until it is assured that any condensate collected in the low places of the smaller line is removed and that the gas in the smaller line is displaced by that from the main. When sampling a gas well that is closed in, the well should be opened to the air or to the line for at least one-half hour before the sample is taken. The location of the well, date, number of well, open-flow capacity,[1] rock pressure, and depth to sand, should be entered in the note-book (see also the *Appendix*, page 541).

It is often necessary to secure samples from lines in which the pressure is below that of the atmosphere. Several forms of apparatus have been devised for this purpose, among which the improved form of the appliance of Hays is said to give good results. The construction of this apparatus is such that water from one of two communicating compartments is forced into the other by means of a hand-pump, the water pumped out being displaced by gas from the line.

2. Density ("Specific Gravity"). (*a*) Effusion Method.—The principle of this method is the comparison of the rate of flow of equal volumes of two gases through an orifice, that of one of the gases being taken as unity. In the natural gas industry it has been customary to use air as the standard.

$$\text{Specific gravity (air = 1)} = \frac{(\text{time for gas})^2}{(\text{time for air})^2}.$$

Apparatus.—Bunsen effusion apparatus (see Fig. 91-d); thermometer; and stop-watch.

Procedure.—Fill the cylinder with water at the room-temperature. Insert the glass tube into the rack with the valve closed,

[1] The open-flow capacity of a gas well is the quantity of gas which will issue therefrom in 24 hours when retarded only by atmospheric pressure.

and place the tip, which contains the platinum orifice plate, in its seat at the top of the tube. Suspend the thermometer in the water.

With the stop-watch in hand, open the valve, permitting the air to escape through the orifice and the water to rise in the tube. Note the time (in seconds) necessary for the water to rise from the lowest mark to the uppermost one below the surface of the water. Record this datum and also the temperature of the water.

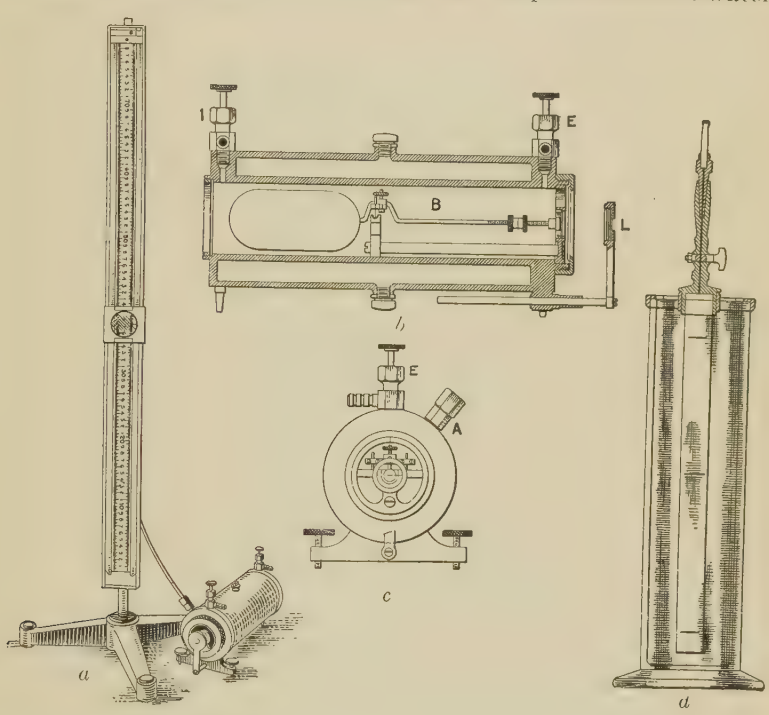

FIG. 91.—Apparatus for the determination of the density of gases.
a—The Edwards gas density balance.
b—Vertical transverse cross-section of the beam and case of *a*.
c—Longitudinal cross-section of the beam and case of *a*.
d—The Bunsen effusion apparatus, sometimes called the "specific gravity tester."

Fill the glass tube entirely with water by suction applied to the top of the tube through a rubber connection, any water collecting in the rubber tube being returned to the cylinder.[1] Next fill the

[1] The use of Bunsen effusion apparatus provided with a three-way valve makes it more practicable to introduce the sample directly and to allow the gas to bubble up through the cylinder for a short time before closing the valve.

glass tube with the gas to be tested, by connecting the rubber tube to the source of supply, over the metal seat at the top of the tube, and opening the valve. When the tube is filled with the gas, the valve is closed, and the apparatus is allowed to stand for a minute before proceeding with the test, as in the case of air. The time in seconds for the water to pass from the lower to the upper mark is recorded, as well as the temperature of the water.

Finally, the time for air is divided into the time for the gas and the result is squared. This is the specific gravity of the gas, compared with air as unity.

Precautions.—The effusion apparatus is convenient for field work and is perhaps more accurate when standardized against the gas density balance. For the best results with the effusion apparatus, the test should not be taken in the open, especially in windy, damp or cold weather. The tip should be inspected carefully at all times and kept dry and warm. If a series of tests is being made, the temperature of the water should vary but little. No water should be lost from the cylinder or none added, since the hydrostatic pressure must be the same when examining both the gas under test and the standard.

Effusion instruments on the market carry a three-way valve and side-tube for the purpose of introducing the gas sample without removing the tip.

(*b*) Gas Density Balance. *Apparatus.*—Edwards' gas density balance (see Fig. 91-*a*, *b*, and *c*); barometer; calcium chloride tube; and pump for evacuating or applying increased pressure.

Procedure.—According to Boyle's law, the density of a gas is proportional to its pressure; and the buoyant force exerted upon a body suspended in a gas is proportional to the density of the gas and, therefore, to its pressure. Hence, if the buoyant force exerted upon a body is made the same when suspended successively in two gases, the densities of the two gases must be identical at the pressures; or the densities of the two gases at normal pressure are in inverse ratio to the pressures when of equal buoyant force.

The balance case is mounted on a firm table and the manometer attached to *A*. This joint is made gas-tight with packing. The manometer is filled half-full with clean mercury.[1] The water

[1] If mercury contains dust or if its surface is fouled, it generally can be cleaned by allowing it to run through a paper filter having several pin holes in the apex. On the purification of mercury, Dennis' "Gas Analysis," **1913**, 117.

jacket should be filled with water at room temperature or sufficient time should be allowed for the water to acquire that temperature before making a test. The beam then is placed on its bearing and inserted in the gas chamber, the support being held firmly in place by the friction-ring to which it is attached. A pair of tongs is provided to aid in removing the beam support. The beam support should be adjusted to be exactly level; this can

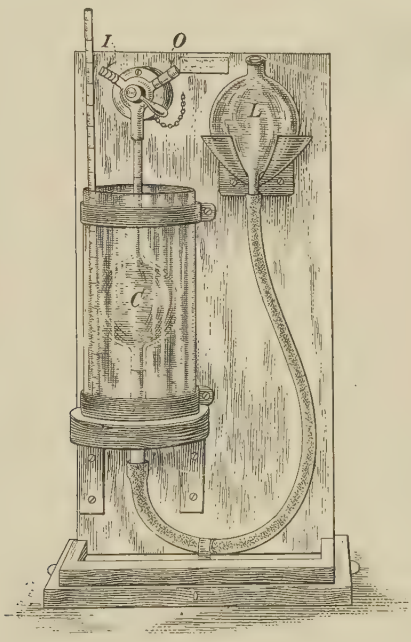

FIG. 92.—The "Standards" specific gravity apparatus for gases, designed by J. D. Edwards (*Met. Chem. Eng.*, **16** (1917), 518).

The effusion method for the determination of the specific gravity of a gas is based upon the fact that the times required for the escape of equal volumes of two gases under the same pressure and through the same small orifice are approximately proportional to the square root of the densities of the gases. This relation is only an approximation, however, and the accuracy obtainable depends on the size and shape of the orifice and the operating conditions. The orifice on the "Standards apparatus" is made in a thin platinum-iridium plate, 0.04 mm. in thickness, and the sharp edged orifice is 0.25 mm. in diameter; this size and shape of orifice has been found by Edwards to be well adapted to this apparatus.

The apparatus consists of a gas chamber (*C*) connected by a rubber tube to a movable reservoir (*L*) which is held at a fixed height in a support as shown. The volume of gas whose effusion time is to be measured is defined by marks on the tubes just above and below the gas chamber. The gas chamber is connected at the top to a three-way cock which allows the gas chamber to be connected with either the gas inlet (*I*) on the left or the tube (*O*) containing the orifice on the right. In brief the method of operation is to measure the time required for a given volume of air to flow through the orifice under the pressure of the head of water in the reservoir and then to measure the time required for the same volume of gas, whose specific gravity is to be measured, to flow through the orifice.

be done by tapping the balance case and observing whether there is a tendency for the beam to slide to either side. The cap clos-

ing the gas chamber then is screwed on tightly and the apparatus is tested for gas tightness. This can be done by reducing the pressure with the vacuum pump to 200 mm. or less, closing the connection to the pump and noting whether the gage shows an appreciable change during a period of five minutes.

The balance case next is filled with dry air by first evacuating and then allowing air to enter slowly through a small calcium chloride tube attached at *I*. A plug of cotton should be placed in the end of the drying tube to prevent dust entering the balance case and becoming attached to the beam. If the balance contains gas from a previous determination, this should be flushed out by evacuating and refilling the requisite number of times to secure an uncontaminated sample. Air should be allowed to enter until the pressure is slightly in excess of that necessary to secure a balance. The exact adjustment of the pressure then can be made by withdrawing air through the needle valve *E*, which enables one to change the pressure very gradually or in steps of a few tenths of a millimeter at a time. When equilibrium is reached, the pressure on the manometer is read and the atmospheric pressure is determined from a barometer. The balance case then is filled with the gas whose density is to be determined. After proper purging, the pressure is adjusted in the same manner as with air. The gage should always be tapped just before reading in order to eliminate the effect of the mercury clinging to the glass.

The sensitivity of the beam can be adjusted by raising or lowering the needle points and by raising or lowering the small nut mounted on top of the beam. The longer the period of swing, the greater the sensitivity, and hence the accuracy. This adjustment must, of course, be made before the beam is placed in the balance case. The beam already has been adjusted for high sensitivity and should not be changed unnecessarily. The position of equilibrium of the beam can be altered by means of the lock nuts at the end of the beam. It is convenient for most work to have it balance a few millimeters below atmospheric pressure.

The balance case may be tapped gently in order to start the beam swinging when there is a tendency to stick. If this binding is serious, the needles should be sharpened and the glass bearing examined for imperfections. If found to be scratched, the bearing should be replaced. The failure to secure constant readings

with air or gas usually is due to an imperfection in the needles or bearings. Any fine, sharp needle can be used for this purpose, and each needle always should be examined to see that it is neither bent nor perceptibly rounded at the point. The exact length of the needles should be measured before removing, to aid in adjusting them after replacement.

The method of recording the data and the calculations involved are given in the following example:

Barometric pressure (millimeters)		756.4
Gage readings with air (millimeters)	396.7 390.3	
Pressure (millimeters)		−6.4
Total pressure (millimeters)		750.0
Gage readings with gas (millimeters)	189.3 597.4	
Pressure (millimeters)		+408.1
Total pressure (millimeters)		1164.5
Specific gravity	$\frac{750.0}{1164.5} = 0.6441$	

On the *calculation* of specific gravity from the results of chemical analysis, see page 259.

3. Heating Value.—The heat of combustion of natural gas may be calculated from the results of analysis by multiplying the heating value of each hydrocarbon by the percentage in the gas, adding the results together, and finally dividing the sum by 100 (see page 260). The reliability of this method depends upon the accuracy of the analysis. In many cases the requisite apparatus or expert analyst is not available, and then the calorimeter offers a fairly reliable method, with the advantage of being operated easily by any intelligent person after instruction and practice.

The apparatus described below is the improved type of Junkers flow calorimeter, which is used very widely in the natural and manufactured gas industries.[1]

Apparatus.—Calorimeter complete (see Fig. 93) and barometer.

Procedure.[2]—On unpacking the calorimeter, see that all parts are present and in good condition. Clean it inside and outside

[1] For a description of the method of the Bureau of Standards, see the *Appendix*, page 503.

[2] From *Report* of the American Gas Institute, with *addenda*.

and be sure that it is free from all packing material. Set up the apparatus as shown in cuts of the different sets. Screw on the inlet water pipe and see that the air vent tube is in its place in this pipe. Level the calorimeter by means of the screw feet and plumb-bob. Connect the center hose nipple on the inlet weir with rubber tubing to the water supply and the side connection to the sink to carry away the over-flow. Connect the tubing for water running to weighing pail to the vertical nipple on the 3-way cock on the outlet weir and for the waste to the side nip-

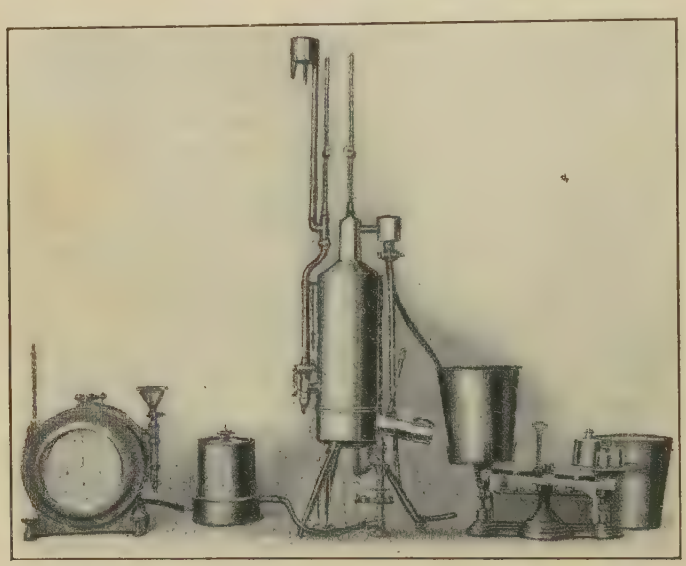

Fig. 93.—Gas calorimeter of the flow type.

ple. Handle the thermometers with the greatest care. Screw the 32° to 100° thermometer on the inlet water pipe and the 60° to 110° thermometer on the top of the instrument for the outlet water. Screw the small thermometer in place on the exhaust flue. Place the two telescopic sights in position on the water thermometers, being very careful not to break them off by pressure against the sights. Connect the meter to the regulating manometer or governor and the governor to the burner with short pieces of rubber tubing, or with flexible metal tubing having coupled ends.

The calorimeter should be set up in a light and well ventilated room or cabinet which is free from draughts and in which the

temperature can be maintained constantly at not less than 60°F. The room should be provided with a sink and with a good supply of running water. It is advisable to have a large shallow overhead covered tank, from which the water supply can be taken. If the tank does not hold enough water for a prolonged series of readings, a small gas water heater may be employed to bring the water to approximately the room temperature. It is desirable to use in the calorimeter water which is clear and free from suspended matter; therefore, a filter should be installed in the water supply line to the overhead tank.

If only a single test is desired, gas may be taken from the house piping; but if an average value is required, a small gas holder, or averaging tank, should be used, and the gas flowing into the holder adjusted to a rate of flow to just fill it in the time during which the sample is to be taken. Care should be exercised to have a short service to this holder in order that an average sample of gas may be obtained; and if the sample is taken from a line on which there is no considerable consumption, it is necessary that this line be thoroughly cleared out before sampling. It is recommended that the gas be metered at a pressure not to exceed 2 in. of water; if this pressure is not obtainable, it is advisable to insert a holder or a diaphragm governor in the supply line to reduce the pressure to this range.

Set up the calorimeter so that the overflow and outlet water can be led to the sink. Make the water connections with rubber tubing, being careful not to cramp the tubing. To avoid air currents caused by the movement of the observer's body, set up the calorimeter so that the water supply and waste may be adjusted easily and that all temperatures may be observed readily. Lead the outlet water to a waste funnel supported a little above the top of the copper or glass container used in collecting the water, so that the water can be shifted from the funnel to the container and back without spilling.

Set up the gas meter facing the observer and level it carefully. Then adjust the water level of the meter, both inlet and outlet being open to the air. To do this, remove the plug from the dry well, open the funnel cock and disconnect the tubing on the outlet of the meter. Add water through the funnel or remove by the cock under the gage glass until the lowest edge of the meniscus just touches the scratch on the gage glass, or is even with the fixed marker. If the meter has been filled freshly with

water, the gas must be allowed to burn at least two hours before making a test. When the water in the meter is saturated with gas, 20 minutes should be sufficient.

Fill the pressure regulator about ¾ full with water, then connect it to the calorimeter burner. Metallic tubing is preferable; but when rubber tubing is used to connect the meter, pressure regulator and burner, the connections should be as short as possible and should be saturated with the gas. Turn on the gas and allow it to burn for 5 to 10 minutes, with the burner on the table. Shut off the gas at the burner and watch the hand on the meter for leakage. Be sure that all leaks are stopped before attempting to make a test. Start the water running through the calorimeter at a rate of about 3 pints per minute. Regulate the gas to flow at the rate (4 to 7 cu. ft. per hour) found by experiment to give the highest result with the gas to be tested, admitting sufficient air through the burner to give the flame a faintly luminous tip; then insert the burner as far up into the combustion chamber as the bracket permits, and again observe the condition of the flame to see that it is all right, using a mirror.

The excess of air passing through the calorimeter is controlled somewhat by the position of the damper in the exhaust port, and the best results are obtained by having the excess air as low as possible and still maintaining complete combustion of the gas. To determine this position of the damper, some experimentation may be necessary. Operate the calorimeter until a thermal balance is established on the inlet and outlet water thermometers. Start with the damper closed, then open slightly, observing carefully the outlet thermometer. When this thermometer reads at a maximum—or, in other words, when the greatest rise in temperature is given to the water, which presumably is passing through the calorimeter uniformly—the damper is in approximately the correct position for the amount of gas being burned and the excess air necessary for perfect combustion is at a minimum.

The water should be regulated so that there is a difference between the inlet and outlet temperatures of about 15°F. The temperature of the inlet water should vary but little when an overhead tank is used and the water maintained at room temperature. Be sure that both overflows are running.

Before making the test, the barometric reading, the tempera-

ture of the gas at the meter, the temperature of the room and the temperature of the exhaust products should be recorded. It is desirable to have the temperature of the inlet water and the temperature of the exhaust products as nearly as possible at room temperature, in order to establish more nearly a thermal balance —the difference in these temperatures should never exceed 5°F. Next allow the gas to burn in the calorimeter until a thermal balance is established, or until there is the least change in the inlet and outlet waters.

The test now may be started by shifting the outlet water by the three-way cock from the funnel to the container just as the large hand on the meter passes the zero point. Readings then are made on the inlet and outlet thermometers, making the readings as rapidly as the observer is able to record them during the consumption of 0.2 cu. ft. of gas. At least ten readings should be made of both the inlet and outlet water temperatures. Water again is shifted from the container to the waste funnel as the hand passes the zero point the second time. The water then is weighed or measured. The uncorrected heating value per cubic foot is obtained by multiplying the difference of the averages of the inlet and outlet temperatures by the number of pounds of water and dividing by two-tenths. This quantity is divided by the correction factor for the barometer and temperature, obtainable from tables, to give the heating value at 30 in. pressure and 60°F. The weight of the contents of the container should be obtained while the inside is wet. This may be done by filling the container with water, emptying, and then shaking for about 5 seconds in an inverted position. This will do away

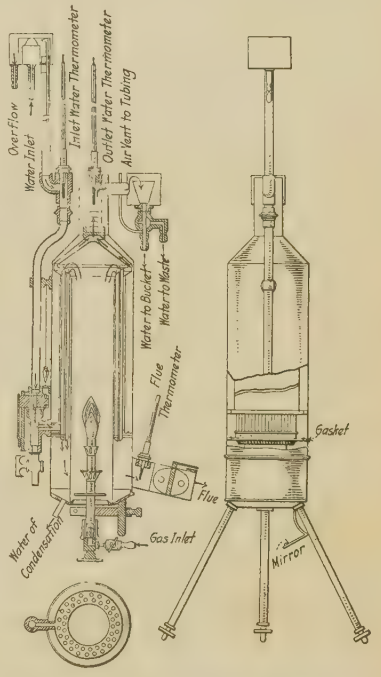

FIG. 94.—Gas calorimeter of the flow type (sectional view).

with any correction where several consecutive tests are required with the same container.

A second, and perhaps a third, test is advisable, and should be made without disturbing the existing conditions, provided all readings are within the above prescribed limits. In practice the operator should get consecutive results within ten (10) B.t.u. on the same holder of gas. Under such conditions an average of the results may be taken.

Results as Obtained by Calculation.—The method of calculating the calorific value of the gas from the observations indicated is very simple when all readings are made in English units, as recommended, and entered in a conveniently arranged form. A simple record sheet is shown in the *Appendix*, page 531.

The averages of the inlet and outlet water temperatures are determined and necessary corrections for thermometer errors are made. The difference in these averages should give the rise in temperature of the water. This rise in temperature is multiplied by the number of pounds of water passed through the calorimeter during the test. The product then is divided by the quantity of gas burned—0.2 cu. ft. This quotient will give the heating value of 1 cu. ft. of gas in B.t.u. at the indicated temperature and barometric pressure. To correct this to 60°F. and 30 in. pressure, divide by the "Correction Factor" for the indicated temperature and pressure as obtained from the *Appendix*, page 527. The final result will be the corrected heating value of the gas tested, in B.t.u.

Expressing the above in a formula, there is had:

$$\text{B.t.u. per cu. ft.}^{1} = \frac{W \times T}{G}.$$

W = weight, in pounds, of water passed.

T = the average difference in temperature, in degrees F., between inlet and outlet water.

G = corrected volume of gas burned, in cu. ft.

Finally, if so desired, the correction for atmospheric humidity is made.

Care of Instruments.—The calorimeter, being a delicate and sensitive instrument, should be cared for when not in use. If the instrument is set up permanently, provision should

[1] Under the following conditions: 30 in. of mercury, 60°F., and saturated with water vapor.

be made that it be not disturbed by anybody except the operator. If the instrument is not erected permanently, when dismantled it should be cleaned carefully inside and out, and the thermometers removed and carefully packed in cotton.

It is advisable to inspect the calorimeter bimonthly, to see whether the interiors of the combustion chamber and of the tubes are free from carbon deposits and corrosion. If it is found to be unclean, the interior of the combustion chamber should be washed or wiped out and the tubes should be cleaned. The latter may be freed of any deposit by using a wire brush cleaning rod, similar to that employed in cleaning rifle barrels.

It seems hardly necessary that instructions should be given for the care of such an instrument, but certain precautions should be noted.

Precautions.—Do not place a lighted burner in the calorimeter when water is not running through the instrument.

Do not shut off the water while the gas is burning; but if the water is accidentally shut off, then shut off the gas quickly, to avoid breaking the thermometers.

Do not move suddenly near the instrument during the progress of a test. Slight drafts thus caused will vary the outlet readings and vitiate the test.

Do not fail to check daily the water level in the gas meter.

Do not forget to test the meter and all connections daily for leakages.

Do not erect the calorimeter too close to any heating or lighting appliances, where radiant heat might affect the readings.

Do not make the test with the inlet water temperature over 5 degrees above or below the temperature of the room.

Do not fail to fill the overhead tank with water when through testing, so that it will be ready for the next test.

Accuracy of Meter.—The accuracy of the meter should be known and corrections to the observed gas volumes should be made when necessary. In calibrating the meter, the method described in the *Appendix*, page 532, may be used.

Correction for Humidity.—The correction for humidity may be secured from the *Appendix*, page 529, after having determined the relative humidity of the atmosphere by the following method.[1] A good ventilation is necessary for the success of this experiment. The apparatus used is the sling psychrometer (Fig. 95).

[1] U. S. Dept. of Agriculture, *Weather Bureau Bull.* **235.**

A small rectangular piece of muslin, wide enough to go one and one-third times around the bulb, and long enough to cover it and that part of the stem below the metal, is wetted and wrapped about the lower of the two thermometers, and tied with a thread, first at the top and then at the bottom, in a way which draws the muslin cover neatly over the bulb. The portion below the lower knot is cut off with a pair of scissors. The lower bulb then is dipped in water and the instrument is whirled rapidly for fifteen to twenty seconds, stopped and the temperatures read quickly, the wet bulb thermometer first. This procedure is repeated until the temperature of the wet bulb becomes constant. This temperature is recorded as the wet bulb reading; that of the other thermometer as the air temperature. The relative humidity then is calculated from the curves of the *Appendix*, page 502. Example: dry bulb reading, 80°F.; wet bulb reading, 70°F.; relative humidity, 61.

FIG. 95.—Sling psychrometer.

Note.—Concerning the manipulation of the psychrometer, U. S. Bureau of Standards *Circular* **48** directs as follows: "The psychrometer should be whirled with a steady uniform motion, keeping the hand nearly stationary, between two and three revolutions per second giving a satisfactory rate of ventilation. The stopping of the instrument is the most difficult part of the operation. This can and should be done in a single revolution. By suddenly allowing the hand to follow the instrument up and out into a larger circular or spiral path, the instrument can be brought to rest as it descends, and the wet bulb reading noted within a second or two."

Total (Gross) and Low (Net) Heating Values of Gas.—By the procedure given above for the determination of the heating value, the result secured is the total ("high") or gross value. The low or net value, which often is used by engineers when considering practical combustion, is the one which takes into consideration the heat carried off by the uncondensed moisture. In the calorimeter this heat is given up by the condensation of the water. In order to calculate the lower heat value of the gas, the number of c.c. of water accumulating in the small glass cylinder

beneath the calorimeter by the combustion of 1 cu. ft. of the gas, is measured. This result multiplied by 2.3 gives the value which is to be subtracted.

4. Analysis of Natural Gas.—Natural gas consists principally of paraffins, and methane generally is the preponderating constituent, the characteristic hydrocarbon. Natural gas may contain, besides the hydrocarbons, nitrogen, carbon dioxide, small quantities of argon, neon and helium,[1] and hydrogen sulphide.[2] For the composition of various gases, see the *Appendix*, page 500.

[1] On the application of the interferometer to gas analysis, and especially to the analysis of helium—nitrogen—methane mixtures, see EDWARDS, *Chem. Met. Eng.*, **21** (1919), 560. On helium in natural gas, see *Kan. Univ. Geol. Survey*, **9** (1908), 228. On the helium content of the various natural gas supplies within the British Empire, see *Bulletin* **31**, Mines Branch, Canada Dept. of Mines, 1920.

[2] Hydrogen sulphide sometimes is found in natural gas and may be tested for qualitatively by the use of lead acetate paper. Experts of the Bureau of Mines determine hydrogen sulphide in natural gas by absorption in a standard solution of iodine and titration of the excess of iodine with standard sodium thiosulphate solution.

The Bureau of Mines finds that hydrogen is not present in natural gas. If it is desired to test for that gas, the ordinary method of passing the gas mixed with air over palladium asbestos, or of absorption in colloidal palladium solution (Bureau of Mines *Bulletin* **42**), may be depended upon to give accurate results.

Since the use of alkaline pyrogallate introduces a slight error, especially when the higher paraffins are present, the following method is recommended where small quantities of air are suspected in natural gas (Bureau of Mines *Bulletin* **42**): About 15 c.c. of a ten per cent. solution of sodium hydroxide and 25 c.c. of a 10 per cent. solution of manganese chloride are drawn into a 150-c.c. burette provided at the top with a 100-c.c. bulb. Then 100 c.c. of the gas sample are drawn into the burette and shaken about 15 minutes with the solutions. Prolonged contact between the gas mixture and solutions is essential for complete oxidation. A few c.c. of a 10 per cent. solution of potassium iodide and a 5 per cent. sulphuric acid solution are drawn into the burette. The mixture then is transferred to a flask and immediately titrated with N/40 sodium thiosulphate solution:

$$2MnCl_2 + 4NaOH = 2Mn(OH)_2 + 4NaCl;$$
$$2Mn(OH)_2 + O + H_2O = 2Mn(OH)_3;$$
$$2Mn(OH)_3 + 3H_2SO_4 + 2KI = 2MnSO_4 + K_2SO_4 + 6H_2O + 2I.$$

Carbon monoxide likewise is found to be absent in natural gas. However, if it is desirable to make qualitative tests, the following is recommended (Bureau of Mines *Bulletin* **42**): A few drops of blood, either defibrinated steer's blood or blood drawn by pricking the finger, are diluted with distilled water until the solution assumes a buff-yellow tint. A test-tube of 50 c.c.

Credit is due to George A. Burrell and his associates in the United States Bureau of Mines, for the perfecting of existing methods, as well as for the development of new procedures for the analysis of gases.[1]

Bureau of Mines Method. *Apparatus.*—See Fig. 96.[2]

Procedure.[3]—Oxygen or other gas left in the horizontal capillary train from the previous analysis is displaced by drawing a few c.c.of nitrogen— prepared beforehand by passing air into the pipette and absorbing the oxygen by means of the alkaline pyrogallate solution—from the pipette *d* into the burette. This mixture then is allowed to escape into the air.

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capacity is filled with the solution and the gas sample is passed into the solution until it displaces all but 25 c.c. of it. The tube then is shaken gently for ten minutes. Finally, the treated blood is compared with the normal blood solution of the same dilution in another test-tube similar in shape and capacity to that used for the treated blood solution. The presence of carbon monoxide may be detected in the treated sample by a characteristic pink color when as small an amount as 0.03 per cent. is present in the gas.

Carbon monoxide and olefines also react with dilute palladium chloride solution, the palladium separating out as a black cast or as particles suspended in the liquid. In the case of carbon monoxide, carbon dioxide is formed during the reaction. This test is good for gases containing but traces of either carbon monoxide or olefines.

[1] Bureau of Mines *Tech. Papers* **87** and **104.**

[2] R. P. Anderson (*J. Ind. Eng. Chem.*, **11** (1919), 299) recommends the use of a combustion pipette made from "Pyrex" glass. He also describes the use of a device which will enable the operator to know when no further contraction is taking place during the combustion. This consists of a small glass tube fastened in a vertical position near the combustion pipette and connected by means of a rubber tubing to a glass "T" placed in the rubber tubing between the pipette and its levelling bulb. The upper end of the glass tube should extend to the highest point to which the levelling bulb is brought in making an analysis, in order that the mercury may not be driven out of the opening at the top. After the entire gas sample has been passed into the oxygen, the stopcock on the levelling bulb is closed; or, in the absence of a stopcock, a pinchcock may be employed on the rubber tubing between the glass "T" and the levelling bulb. Under these conditions, contraction of the gases in the combustion pipette will cause the mercury to move downward in the small glass tube, and the stopcock or pinchcock should be opened momentarily from time to time to avoid too large a reduction in pressure within the pipette. When no further movement of the mercury in the indicating device is noted, it is evident that the contraction has ceased and that combustion is as near complete as may be expected for the sample.

On the lubrication of stopcocks, see Dennis' "Gas Analysis," **1913,** 115.

[3] Bureau of Mines *Tech. Paper* **87.**

About 100 c.c. of the gas sample then are drawn from the sample container into the burette. The sample is measured in the burette against the pressure existing in the compensating tube by bringing the mercury in the manometer tube exactly to the mark *g*. The sample then is passed successively into the potassium hydroxide solution to remove carbon dioxide, and the alkaline pyrogallate solution to remove oxygen, the burette measurements being made in the same manner as with the original sample.

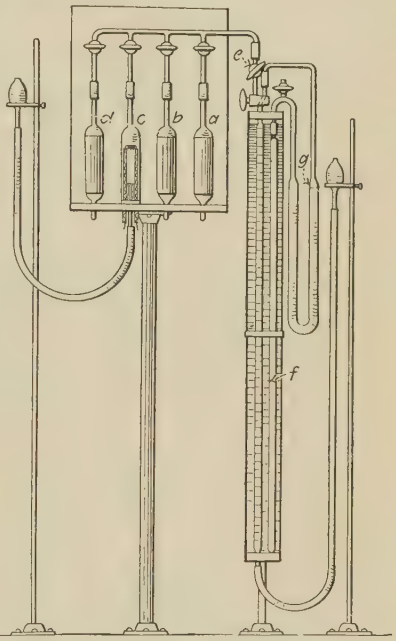


FIG. 96.—Bureau of Mines apparatus for the analysis of natural gas.

At the right are the water-jacketed burette and compensating device. Connected to the first-mentioned are a manifold and the pipettes *a*, (KOH solution), *b* and *d* ("pyro" solution), and *c* (combustion pipette).

The residual gas left after the carbon dioxide and oxygen have been determined is discarded. The capillary connections are cleared of combustible gas by dilution with air, and 100 c.c. or slightly less of oxygen are measured into the burette and passed into the combustion pippette. About 35 c.c. of the gas sample then are drawn into the burette from the sample container and measured. The platinum wire in the combustion pipette is heated electrically to a white heat and the gas sample is passed at the rate of about 10 c.c. per minute into the combustion pipette containing the oxygen. The hydrocarbons burn as fast as they enter, so that an explosion consequent upon the accumulation of gas and oxygen can not follow. In analyzing natural gas, the best results have been obtained by passing the oxygen into the pipette first. When the natural gas is passed in first, the mixture does not always burn satisfactorily.

After the hydrocarbons are burned, which requires about four or five minutes, the combustion pipette is allowed to cool and the contraction in volume due to combustion is measured. The carbon dioxide produced by the combustion is determined by absorption in the potassium hydroxide solution. Then the gas

is passed into the alkaline pyrogallate solution, to make sure that sufficient oxygen has been present for the complete combustion of the paraffins. Some samples of natural gas, especially gas from which gasoline can be recovered, may contain such a large proportion of the higher paraffin hydrocarbons that 100 c.c. of oxygen will not be sufficient for the complete combustion of the 35 c.c. of natural gas. In such instances a smaller quantity of the natural gas sample must be used.

In no case is the mercury in the burette raised above the upper stopcock. The gas remaining in the capillary tubing at any stage of the analysis is brought in contact with the solutions by

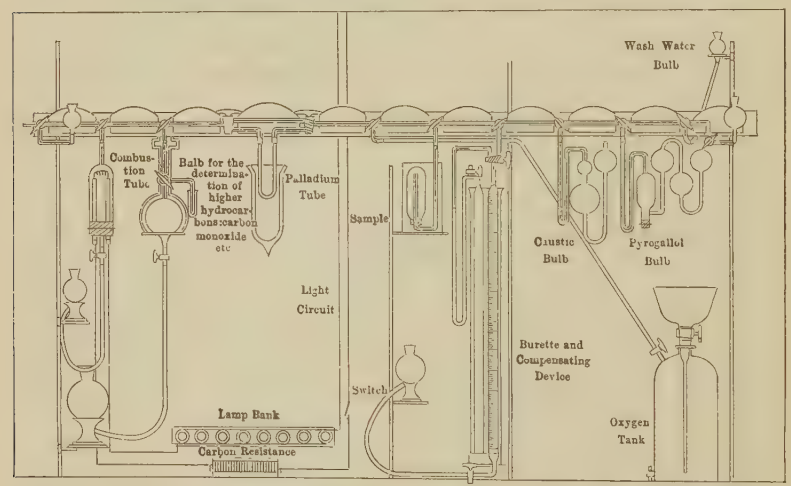


FIG. 97.—The gas analysis apparatus used by Allen and Lyder (*Bull. Univ. of Kan.*, **19**, No. 1; *Bull.* No. **3**, *Div. State Chem. Res.*).

passing it back and forth several times between the burette and the pipette. After combustion, for instance, some carbon dioxide will remain in the capillary tubing between the combustion pipette and the burette when the gas is drawn back into the burette to record the contraction in volume. After most of the carbon dioxide has been absorbed by the potassium hydroxide, the small quantity that remains in the capillary tubing is swept out of the tubing into the potassium hydroxide pipette, and the operation is repeated in order to insure the complete removal of the carbon dioxide. If this precaution is not taken, an error of considerable magnitude will result. Finally, the residual gas is burned again in the combustion pipette, and the amount of

contraction and the volume of carbon dioxide are measured. This second combustion is often necessary in order to burn the natural gas completely.

Example of a Typical Analysis.—A typical analysis with the calculations from the analytical data is given herewith.

First Sample

	Burette readings, c.c.
Volume of sample taken for absorption	30.70
Volume after CO_2 absorption	30.70
CO_2 absorbed	0.00
Volume after O_2 absorption	30.70
O_2 absorbed	0.00

Fresh Sample

Volume of oxygen taken for combustion	74.85
Volume of sample taken for combustion	30.70
Total volume	105.55
Volume after burning	42.30
Contraction	63.25
Volume after CO_2 absorption	7.20
CO_2 contraction	35.10

Methane (CH_4) and ethane (C_2H_6) are calculated from the following theoretical equations:

$$CH_4 + 2O_2 = CO_2 + 2H_2O;$$

$$C_2H_6 + 3.5O_2 = 2CO_2 + 3H_2O.$$

Let x = methane and y = ethane. Then $2x + 2.5\,y$ = total contraction, and $x + 2.0\,y$ = CO_2 produced; whence CH_4 = 84.1 per cent. and C_2H_6 = 15.1 per cent.

The complete analysis of the natural gas is as follows:

Carbon dioxide (CO_2)	a.0[1]
Oxygen (O_2)	a.0[1]
Methane (CH_4)	84.1
Ethane (C_2H_6)	15.1
Nitrogen (N_2)	b.8[2]
	100.0

[1] a = less than 1 per cent.

[2] b = difference.

Calculation of the Specific Gravity.—The specific gravity is calculated from the known specific gravities of the constituents that comprise the natural gas. These are as follows:

Constituent	Specific gravity (air = 1).
Methane	0.5545
Ethane	1.0494
Nitrogen	0.9674
Carbon dioxide	1.5291
Oxygen	1.1054

The specific gravity of the natural gas sample becomes:

$0.841 \times 0.5545 = 0.456$
$0.151 \times 1.0494 = 0.158$
$0.008 \times 0.9674 = 0.007$

Total.....0.621

Calculation of Heating Value.—The heating value of natural gas is calculated by using the heating values of the combustive constituents.

Gross Heating Values of Constituents of Natural Gas

Constituent	Heating value in B.t.u. per cu. ft. at 760 mm. pressure	
	0°C.	60°F.
Methane	1065	1008
Ethane	1861	1761

The gross heating value of the natural gas sample at 0°C. and 760 mm. pressure is, then, as follows:

$0.841 \times 1065 = 896$ B.t.u.
$0.151 \times 1861 = 281$ B.t.u.

Total.....1177 B.t.u.

Reagents.[1] Alkaline Pyrogallol Solution.—Ten g. of pyro-

[1] Jones and Meigham (*J. Ind. Eng. Chem.*, **11** (1919), 311) have investigated sodium pyrogallate as an absorbent for oxygen and recommend the following for use in pipettes having glass tubes on the inside: Stick sodium hydroxide is dissolved in an equal weight of water, and constitutes the stock sodium hydroxide solution. In another container stock "pyro" solution is made up in proportions of 1 g. of "pyro" to 3 c.c. of water. When ready for use, 5 parts of the sodium hydroxide solution are mixed with 2 parts of "pyro."

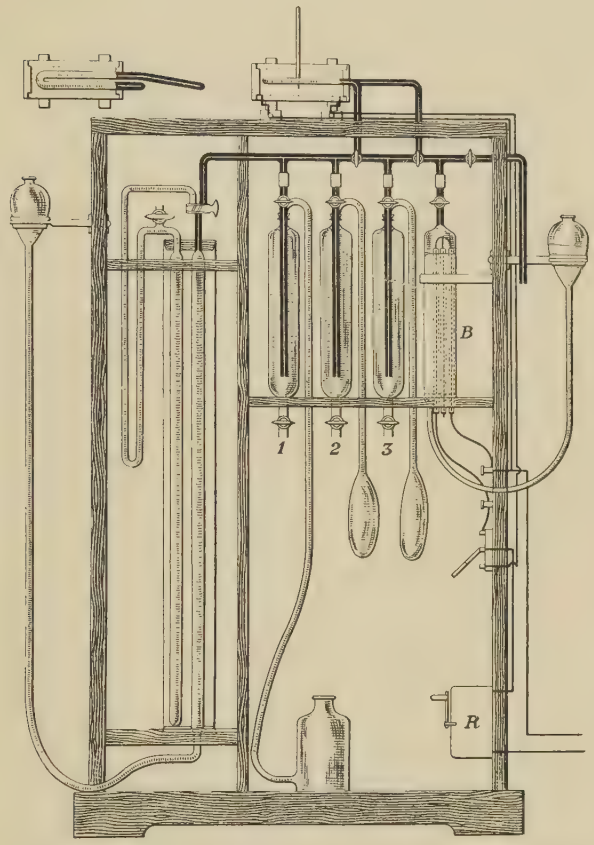


FIG. 98.—The apparatus used by J. G. Davidson for the analysis of the gas produced by cracking the ethane-propane fraction of natural gas condensate (*J. Ind. Eng. Chem.*, **10** (1918), 903). Nothing new is claimed for this modification of Burrell's apparatus (*J. Ind. Eng. Chem.*, **4** (1912), 296), except its greater accessibility and ease of manipulation. Babb pipettes with an extra stopcock blown in the bend, as shown, were substituted for the Ostwald pipettes in Burrell's apparatus; this extra stopcock facilitates refilling and cleaning the pipettes without the necessity of disconnecting from the main part of the apparatus. Beyond this, the form of the Babb pipette lends itself admirably to rapid and complete absorption. The slow-combustion pipette (*B*) was made of transparent quartz, in order to reduce breakage. *R* is an adjustable rheostat in circuit with the double heating wire in the slow-combustion pipette. Pipettes 1 and 2 contained 30 per cent. potassium hydroxide solution, while pipette 3 was filled with saturated bromine water. The unsaturated hydrocarbons were determined by absorption in pipette 3, one passing of the gas being sufficient in cases where the olefine content of the gases was below 17 per cent.

gallol are dissolved in 15 c.c. of water; this solution then is mixed with 100 c.c. of (purest; not "purified by alcohol") potassium hydroxide solution (15 parts of KOH dissolved in 10 parts of water). The solution should be tested frequently with air in order to know that the strength has not fallen to the point where absorption is slow or incomplete.

Potassium Hydroxide Solution.—A solution of one part of potassium hydroxide in two parts of distilled water.

Burrell and Oberfell[1] have described procedures for the analysis of gases containing olefines, carbon monoxide, hydrogen, paraffins, carbon dioxide and nitrogen.

Calculations from Combustion Data.[2]—If the contraction and the volume of carbon dioxide from the combustion of the gas indicate methane only, this constituent is reported. But if the data indicate higher members of the paraffin series, a calculation is made that gives the two predominating constituents. The gaseous hydrocarbons react with oxygen as follows:

$$CH_4 + 2O_2 = CO_2 + 2H_2O;$$
$$C_2H_6 + 3.5O_2 = 2CO_2 + 3H_2O;$$
$$C_3H_8 + 5O_2 = 3CO_2 + 3H_2O;$$
$$C_4H_{10} + 6.5O_2 = 4CO_2 + 5H_2O.$$

But since the experiments of Rayleigh, Leduc, Baumé and Perrot, and others, have shown that certain gases deviate from the gas laws, corrections must be made in the case of some gas analyses where this deviation is greater than the experimental error.

The following is a table of the theoretical and observed specific gravities of the gases involved in the calculations, taken from Landolt and Börnstein.[3]

[1] *J. Ind. Eng. Chem.*, **8** (1916), 228; see also Davidson, *ibid.*, **10** (1918), 902 (see page 261).

[2] Bureau of Mines *Bulletin* **42.**

[3] "Physikalisch-chemische Tabellen," **1912,** 148.

THEORETICAL AND OBSERVED SPECIFIC GRAVITIES OF CERTAIN GASES[1]

Gas	Molecular weight	Specific gravity		Observer
		Theoretical	Observed	
CH_4	16.03	0.5538	0.5545	Baumé and Perrot
C_2H_6	30.05	.....	1.0494	Baumé and Perrot
C_4H_{10}	58.05	2.0065	2.01	Frankland
CO	28.00	0.9673	0.96702	Leduc
CO	28.00	0.9673	0.96716	Rayleigh
CO_2	44.00	1.5201	1.52874	Leduc
CO_2	44.00	1.5201	1.52909	Rayleigh
N_2	28.02	0.9680	0.96737	Rayleigh
N_2	28.02	0.9680	0.96717	Leduc
O_2	32.00	1.1055	1.10535	Rayleigh
O_2	32.00	1.1055	1.10523	Leduc

[1] At 0°C. and 760 mm. pressure.

Below are given the equations for the reactions of methane and ethane with oxygen, in which corrections have been made according to the specific gravity determinations shown in the preceding table.

$$0.999\ CH_4 + 2.000\ O_2 = 0.994\ CO_2 + 2H_2O;$$
$$0.990\ C_2H_6 + 3.500\ O_2 = 1.988\ CO_2 + 3H_2O.$$

As the partial pressure of gases in a mixture decreases, the gases more nearly conform in behavior to the gas laws; consequently, a table is presented which shows the correct molecular volume to use in the case of carbon dioxide for different partial pressures. The partial pressure of the carbon dioxide has reference to the ratio of the volume occupied by the carbon dioxide found after the combustion to the total volume of the residual gas observed after the combustion; that is, if the total volume after combustion is found to be 70 c.c., and the carbon dioxide is 40 c.c., then the partial pressure of the carbon dioxide will be $\frac{40 \times 760}{70} = 434$ mm., and the proper molecular volume, namely, 0.997, will be found from the table.

The foregoing equations have reference to their use at 0°C. and 760 mm. pressure. The following table shows the correct molecular volumes for carbon dioxide at 20°C. and different partial pressures.

In compiling this table advantage was taken of the work of Rayleigh and of Leduc and Chappius having to do with the determination of the specific gravity and coefficient of expansion of carbon dioxide. The specific gravity determinations were given by Rayleigh and Leduc for carbon dioxide at 0°C. and 760 mm. pressure. Values for 20°C. and 760 mm. pressure were determined from the coefficient of expansion of car-

MOLECULAR VOLUME OF CARBON DIOXIDE CORRESPONDING TO DIFFERENT PARTIAL PRESSURES AT 20°C.

Mm. of mercury	Molecular volume
100	0.9993
200	0.9986
300	0.9980
400	0.9972
500	0.9965
600	0.9958
700	0.9951
760	0.9950

bon dioxide between 0°C. and 20°C. A graph was plotted from two values, the deviation from the gas laws at 760 mm. pressure and at 380 mm. pressure.

The coefficient of expansion of ethane between 0° and 20°C. has not been reported; consequently, the same molecular volume was used at 20°C., the laboratory working temperature, as was found by Baumé and Perrot[1] at 0°C. The error resulting from this usage can be disregarded without introducing any appreciable error in the analyses, judging from the molecular volume of carbon dioxide, which, at 20°C., is only 0.001 different from the value at 0°C.

Below are given the molecular volumes to be used in the case of ethane for different partial pressures.

MOLECULAR VOLUME OF ETHANE CORRESPONDING TO DIFFERENT PARTIAL PRESSURES

Mm. of mercury	Molecular volume
0	1.000
100	0.999
200	0.997
300	0.996
400	0.995
500	0.993
600	0.992
700	0.991
760	0.990

Although the individual paraffins in a mixture of several cannot be determined exactly, one will know with a sufficient degree of accuracy which value in the above column to use by accepting the value that corresponds to the percentage of ethane determined from the combustion analysis. It is also true that in natural gas from many places the

[1] See LANDOLT and BÖRNSTEIN'S "Physikalisch-chemische Tabellen," **1912**, 148.

partial pressure of ethane is so low that only a small deviation from the gas laws occurs. A slight error arises from the probable percentage of propane or butane in a mixture when the combustion analysis indicates only methane and ethane. In most cases the partial pressures of the propane and butane also will be so low that errors in molecular volumes due to their presence can be disregarded. Methane conforms so closely to the gas laws that no deviation from the given molecular volume need be made for different partial pressures.

The proper equations to use can be determined only from the partial pressures obtained from the analyses. For the purpose of determining the approximate percentage of ethane the theoretical equations can be used.

Application of the Use of Corrected Equations to the Analysis of Natural Gas and Other Gas Mixtures.—Although the combustion analysis does not show an accurate distribution of hydrocarbons in a natural gas mixture, it does reveal the true total hydrocarbon content. The heating value calculated from such an analysis is also correct.

Determinations by the slow-combustion method show that the natural gas supplied to Pittsburgh from the Appalachian field contains about 83 per cent. of CH_4, 16 per cent. of C_2H_6, and 1 per cent. of nitrogen. The proportions vary from time to time during the year. A typical analysis, with the calculation from the analytical data, is given herewith.

TYPICAL ANALYSIS AND CALCULATION

	Burette readings (c.c.)
Sample taken	30.70
Volume after CO_2 absorption	30.70
Portion taken for combustion	30.70
Oxygen added	74.85
Total volume	105.55
Volume after burning	42.30
Contraction	63.25
Volume after CO_2 absorption	7.20
Carbon dioxide	35.10

CH_4 and C_2H_6 are calculated from the theoretical equations as follows:

Let x = methane,
and y = ethane;
then $2x + 2.5y$ = total contraction,
and $x + 2.0y$ = CO_2 produced.
C_2H_6 = 15.1 per cent.
CH_4 = 84.1 per cent.

Total paraffins = 99.2 per cent.

They are calculated from the corrected equations as follows:

Let x = methane,
and y = ethane;
then $2.004x + 2.5y$ = total contraction,
and $0.996x + 2.0y$ = CO_2 produced.
C_2H_6 = 15.7 per cent.
CH_4 = 83.1 per cent.

Total paraffins = 98.8 per cent.

Analysis of Natural Gas by Fractional Distillation at Low Temperatures and Pressures.—The analysis of natural gas by the combustion method just given, while satisfactory for most purposes and permitting calculations of heating value and density which are correct, still is inaccurate to the extent that only two of the combustible constituents may be determined—methane and ethane.

A method has been developed by Burrell and his collaborators in the Bureau of Mines,[1] the principle of which is fractional distillation at low temperatures and pressures. The apparatus used includes a Töpler vacuum pump, Dewar bottles, and gas analysis apparatus; the necessary low temperatures are secured by means of liquid air and of solid carbon dioxide.

The method is as follows: (*a*) The methane is separated by fractionating twice at −190°C. (*b*) The boiling-points of ethane and propane are too close together to permit complete fractionation by this method. The procedure here is to secure at −130°C. two fractions, one of which contains all the ethane and part of the propane, the other fraction containing all the butane and a part of the propane. The two fractions, being mixtures of but two components, then are analyzed accurately by the combustion method. (*c*) Finally, the composition of the original is calculated from the respective data.

A comparison of natural gas analyzed by both methods is as follows:

Analysis by Combustion

	Per cent.
Methane	79.2
Ethane	19.6
Nitrogen	1.2

[1] See *Bulletin* **42** and *Tech. Paper* **104.**

ANALYSIS BY FRACTIONAL DISTILLATION AT LOW TEMPERATURES AND PRESSURES

	Per cent.
Methane	84.7
Ethane	9.4
Propane	3.0
Butane and higher	1.3
Nitrogen	1.6

5. Gasoline in Natural Gas.—In testing natural gas for gasoline content, various laboratory methods have been devised which are reliable when employed by those thoroughly experienced in their use and familiar with the particular field under consideration.[1] Before installing plants, however, it is customary to make practical field tests, using either a small compressor or an absorber, or both.

In the case of "rich" gases which are to be treated by compression, a compressor capable of delivering a maximum of 200 to 500 cu. ft. of gas per hour at a maximum pressure of 250 to 300 pounds to the sq. in. (see Fig. 99) generally is used in preliminary tests. The discharged gas from the compressor is led through a coil cooled by water, the condensate collected, measured and tested for Baumé gravity.[2] The gas used in the test is measured

[1] Bureau of Mines *Tech. Paper* **87** gives various methods of determining the gasoline content of natural gas. On "wet" and "dry" natural gas, see BACON and HAMOR, *lib. cit.*, **2,** 864 and 922.

[2] A portable outfit for the testing of natural gas for gasoline content by the compression method, observed in operation by DYKEMA (Bureau of Mines *Bulletin* **151**), was designed as follows: Tank, 12 in. by 36 in., used as a gas receiver; a 4-h.p. gas-engine belted to a 3 by 3½ in. single-acting compressor with a capacity of 3 cu. ft. per minute; a single coil of 1-in. pipe of the continuous return-bend type, cooled by submerging in a wooden trough of water and ice; and a double coil, 12 ft. long, of 1-in. pipe inside of a 2-in. pipe, cooled by expanding the compressed gas through a valve connection between the outside and inside pipes.

The gas was brought from the well line through a ½-in. pipe fitted with a water gage for regulating the pressure on the intake receiver; this receiver also served as a trap for heavy oil or dirt. From the receiver the gas was compressed and delivered to the water-cooled coil at a pressure of 250 lb., the water being cooled with ice. The gas from the water-cooled coil discharged into the chamber between the outer and inner pipes of the double coil. At the discharge end of the outer or 2-in. pipe was suspended a 2-in. drip to collect the condensate. Gas from the 2-in. pipe was expanded through the valve into the inner pipe to refrigerate the high-pressure gas flowing through the outside pipe and discharged through a service meter to the atmosphere. Records were kept of temperatures, pressures, and

by a suitable meter recently tested for accuracy. From the volume of gas used in the test and the volume of gasoline secured, the yield of gasoline in gallons per thousand cu. ft. of gas is calculated. Separate tests should be made at 100, 200, and 250 pounds to the sq. in.

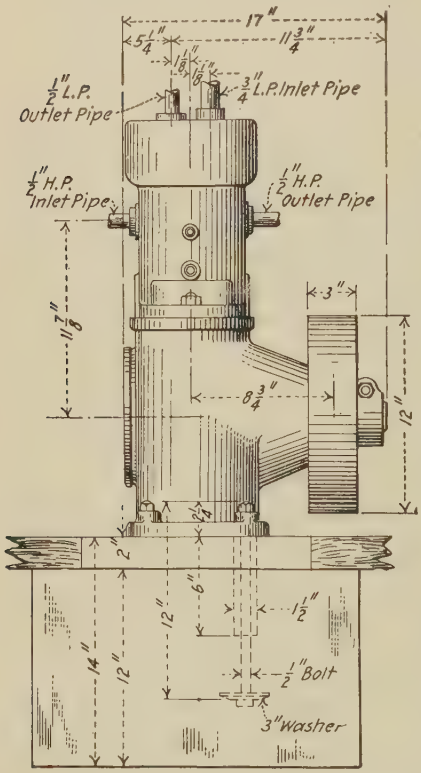


FIG. 99.—Compressor for field compression tests on natural gas.

For the testing of "dry" or "lean" gas some form of portable absorber generally is used. The apparatus (method A) described in the following paragraph is the model of the United States Bureau of Mines, and is to be recommended especially because of its elasticity—that is, one, two, three, or four of the compartments may be filled with the absorbing medium as the occasion demands.[1]

(*a*) BUREAU OF MINES METHOD. *Apparatus.*—See Fig. 100. The apparatus consists of a piece of 6-in. casing with five separate compartments, each of which is connected with a ¾-in. gas inlet, and also a 2-in. gas discharge pipe or separate chamber which extends

amounts and gravity of condensate produced. From time to time specific gravity tests of gas from individual wells were made and recorded. When the gas from any one well dropped noticeably in specific gravity, a compression test was run on it, and, if found too low in gasoline content for profitable recovery, was turned into the fuel lines.

Sources of Error.—The greatest source of error in making tests with portable outfits, is the meters. Some operators use two meters, one on the intake and one on the discharge of the portable tester, the indicated amounts of gas being averaged in calculating the production.

[1] This apparatus also is recommended as giving very satisfactory results with rich gas.

to a point near the bottom of the casing. From the casing runs a ⅜-in. pipe coiled around a 3-in. core with seven turns, through which the gas being treated bubbles and in which most of the absorption takes place. Some small modifications and additions, optional in the design and not shown in the figure, are the use of needle valves at each extremity of the apparatus in order that gas may not be introduced too rapidly or may be throttled to any desired pressure so as not to carry oil over from one compartment to the next; also, the use of a

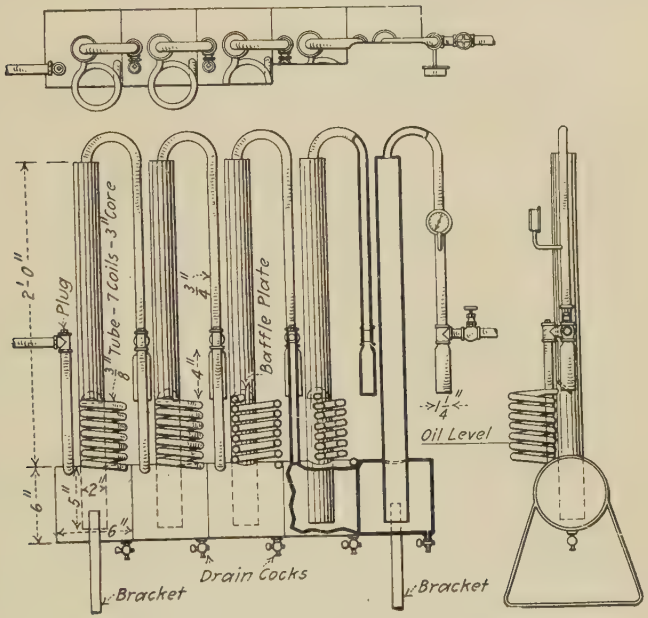


Fig. 100.—Dykema-Neal test absorption apparatus.

needle valve on the discharge end to enable one to regulate more easily the rate of flow through the absorber very slowly. It is advantageous to use gate valves instead of drain cocks for drawing off treated absorption oil from the oil chambers, for such valves facilitate rapid work and eliminate the possibility of volatilization losses when oil is allowed to spray through a stopcock under pressure, into the container for collecting the treated oil. Then, too, time can be saved by using small bull-plugged nipples, in place of standard plugs, since they can be more easily removed and more rigidly connected to prevent leaks.

Procedure (after Dykema and Neal[1]).—To make the test with this absorber, 2700 c.c. of mineral seal oil, or *enough* to bring the level of the oil about 2 in. *above* the top of the 6-in. casing and well above the coil inlet, are measured accurately and introduced into each compartment. The most important requisite for absorption media is high initial boiling-point, in order that in the subsequent distillation a quantitative separation can be effected. The oil used in the tests described by Dykema and Neal had the following physical properties:

Baumé gravity	36°
Initial boiling-point	450°F.
Viscosity (Saybolt universal instrument, at 100°F.)	51

In most tests only the first three absorbers are used, but it may be expedient to fill the fourth compartment when examining rich gases at low pressures or when running a large volume of dry gas in paralleling a 12-hour or day's operation of an absorption plant to determine the extraction efficiency. The fifth division is not intended to be used as a container for oil, but to serve as a separator or oil trap in case any oil is carried over from the preceding compartment. A meter capable of measuring accurately from one to 1000 cu. ft. of gas per hour is connected to the discharge of the absorber.

The gas to be tested is allowed to enter the apparatus slowly with the discharge valve closed, and, when pressure equilibrium with the gas to be examined is obtained, the discharge valve is opened enough to permit the desired rate of flow through the meter. The gas entering the absorber bubbles up through the oil, the latter absorbing the gasoline. The function of the pipe coil is to provide long and intimate contact between the oil and the gas as the gas passes through the absorber. After the desired quantity of gas has passed through the absorber, the supply is shut off and the pressure is released through the needle discharge valve, allowing all the gas to flow through the meter. After the pressure has fallen to atmospheric pressure, all the oil is withdrawn at the bottom of the casing and the oil from each compartment is measured accurately, 1000 c.c. of treated oil from each compartment being kept for the distillation test.

Distillation of Saturated Oil.—400 c.c. of the treated oil are

[1] Bureau of Mines *Tech. Paper* **232**.

introduced into a 500-c.c. Engler distilling flask connected to a condenser made of ½-in. brass tubing and surrounded by ice water contained in a metal box. The flask is heated by direct flame, slowly at first, and the gasoline driven out of the oil is collected in a graduated cylinder. The flask is heated until the vapor reaches a temperature of 350°F., which usually requires 20 minutes. If the oil has a very high saturation, it then is allowed to cool 20° or 30°, after which it once more is raised to 350°F. This procedure is followed until practically no more gasoline is driven over and collected from the condenser.

The extraction of gasoline by the oil will depend upon the rate of flow, the gasoline content of the gas, the volume of gas treated, the pressure, and the temperature of the absorbing oil. The method is best suited for use in testing gas containing 0.5 gallon or less of gasoline per 1000 cu. ft. Optimum conditions as regard volumes of gas and rates of flow with gases at different pressures and of gasoline contents are given below.

Errors in Making Absorption Tests.—R. B. Dow, of the Bureau of Mines, has reported the following:

(1) The saturation of the absorbing oil should be as high as possible in the first compartment and as low as possible in the last. Care should be exercised in admitting gas into the absorber—this should be done slowly, so that oil is not thrown from one compartment to another. It is preferable to distil the oil from each compartment separately; and if the last compartment shows too high a percentage of absorption, to repeat the test. (2) The percentage of gasoline recovered by distillation (dry heat) is dependent upon the saturation of the oil. The lower the percentage of absorption, the lower the recovery of gasoline by the method of distillation. (3) During the distillation the temperature of the oil should not be raised too rapidly. (4) During the distillation the temperature of the condenser should be below 60°F. (5) It is advantageous to stop the distillation just before the mineral seal "ring" reaches the thermometer. The mineral seal "ring" is the top of the oil vapors which gradually rise when the boiling-point of the oil is reached.

In considering the findings of Dow, the logical conclusion is that, in distilling the charged oil, larger aliquot portions, 1000 c.c. or more, should be distilled, the gravity of the distillate determined, and its volume corrected to that of a standard gravity by means of the table on page 280. In using any type of absorbing apparatus it is advisable to experiment and to compare the results obtained with the results from commercial plants of known design, under varying conditions of

pressure and temperature. Curves showing the effect of temperature and pressure on yield are shown in the *Appendix*, page 546.

CONTROLLING FACTORS IN OPERATION OF TEST ABSORBER

Maximum rates of gas, cu. ft. per hour	Pressure, lb. per sq. in.
400	300
200	150
100	75
50	40
20	Atmospheric

MAXIMUM GAS CAPACITY

Cu. ft. of gas	Gasoline, gal. per 1000 cu. ft.
800	0.125
400	0.250
200	0.50
150	0.75
100	1.00
66	1.50
50	2.00
35	3.00
25	4.00

The following data were obtained from a representative test made upon the intake gas at a compression plant in the Mid-Continent field:

INTAKE GAS

Temperature of oil	94°F.
Pressure	204 lb. to the sq. in.
Rate of flow	133 cu. ft. per hr.
Volume	302 cu. ft.

COMPARTMENTS

	1st	2nd	3rd
Charge, c.c	2600.0	2600.0	2600.0
Recovered, c.c	2700.0	2660.0	2630.0
Baumé gravity of oil, degrees	37.0	36.4	36.0
Distilled, c.c	400.0	400.0	400.0
Gasoline, c.c	19.5	9.5	3.0
Total each, c.c	131.5	63.2	23.0
217.7 c.c. (78°Bé.)			
Proportion extracted in each compartment, per cent	60.5	29.0	10.5

Gasoline content: 0.199 gal. per 1000 cu. ft.

The gasoline content is calculated by using the following formula:

$$Q = \frac{100\,C}{G} \times \frac{C}{3785}.$$

Where Q is the gasoline content in gallons per 1000 cu. ft. of gas, G is the volume (cu. ft.) of gas treated, and C is the total number of c.c. of gasoline obtained from the treated absorption medium.

The *Appendix*, page 546, shows the effect of temperature and pressure on absorption.

Notes.—When testing a gas for yield of gasoline by either absorption or compression, it is desirable to distil a sample of the gasoline by the method given on page 56, in order to provide a distillation curve. When testing a source of supply composed of a group of wells feeding into a trunk line, it is advisable to test not only the trunk line but also the individual wells. An individual well which has been closed-in should be vented for at least ½ hour before making a test. In carrying out a test, after the operation is proceeding smoothly, the connections should be examined for leaks by means of soap suds or heavy lubricating oil, and any leaks found should be closed immediately. In considering the installation of a gasoline plant many factors should be weighed, such as the probable life of the field, possibility of extensions, water supply, distance from railroad, cost of gas, effect of reducing pressure on the wells, and type of plant, whether compression, absorption, or combination low pressure and absorption. In *distilling* the charged oil, good results may be had by means of *steam distillation.* A 1-liter sample is placed in a 2,000-c.c. round-bottom flask having a rather long neck. A three-hole stopper accommodates the steam inlet tube, vapor outlet tube and thermometer. The temperature of the oil is raised slowly by means of direct steam and care is taken during the distillation to ensure that none of the absorbing oil is thrown mechanically into the condenser. The condenser should have a *long* jacket and ice-cold water should be circulated continuously during the distillation. The receiver at the outlet of the condenser is surrounded by ice water.

(*b*) Charcoal Absorption Method.—The most recently developed method for the determination of gasoline in natural

gas is that of absorption by charcoal.[1] On account of the simplicity of operation, the method promises to become of importance. The following is a description and discussion of the method by Oberfell and Burrell.[2]

The most practical method heretofore employed for testing natural gas for gasoline content consisted in absorbing the condensable vapors from the gas by means of oil and recovering them by distillation. The method described *infra* depends upon the use of a solid absorbing medium, such as charcoal, and is applicable to both "lean" natural gas, such as is obtained from gas wells, and so-called casinghead gas, which is generally rich in gasoline content.

The absorber is a tin tube, 1⅛ in. in diameter and 10 in. in length. This tube contains a perforated disc near one end. Screw caps fitted with rubber composition gaskets are used to insure air-tight joints. An extra screw cap with a hole at the top is fitted with a short piece of ⅛-in. copper tubing. The receptacle therefore may be used either as an absorber or as a sample container. The equipment case is used for testing wells for volume of gas and for making absorption tests of gas for gasoline. After absorption of gasoline vapors from a measured quantity of gas in the field, the charcoal is sent to the laboratory for distillation, to determine the gasoline content as described below. This equipment case contains thirteen (13) sample containers, filled with charcoal, one orifice meter, one "U" tube, or manometer, and one dry meter (No. 607, American Meter Co., New York). The combined weight of case and contents is 22 pounds.

Approximately 250 c.c. of about 8–14-mesh highly absorbent charcoal, which is practically free from moisture or other foreign absorbent (such as silica gel), is used for each test. A measured quantity of gas then is passed through the charcoal at atmospheric pressure and the sample of charcoal enriched by gasoline vapors is sent to the laboratory, where the test is completed. It is advisable to pass the metered gas first through calcium chloride to remove moisture, unless the charcoal has a very high absorption value, since otherwise the charcoal might absorb enough moisture from the gas to impair its value as an absorbent for the gasoline vapors. The amount of gas that should be passed through the charcoal depends upon the gasoline content. It is advisable to use a volume which will give 10–20 c.c. of gasoline upon dis-

[1] The use of charcoal and activated charcoal in the determination of gasoline in natural gas has been studied in the laboratory of J. B. Garner, at the Mellon Institute. Garner and J. E. Underwood experimented with this method in 1915.

[2] *Oil and Gas J.*, July 11, **1919**, 70. On a portable apparatus using charcoal in testing natural gas for gasoline, see Anderson and Hinckley, *J. Ind. Eng. Chem.*, **12** (1920), 736.

tillation of the enriched charcoal. The specific gravity of the gas will afford some idea of the amount of gas to use for a test.

The rate at which the gas is passed through the charcoal is about 20 cu. ft. per hour, the rate being limited by the capacity of the meter. Tests were made on natural gas with a gasoline content of 250 gallons of 84°Bé. product per million cu. ft. In these tests it was shown that there was practically no difference in the results of tests with the rates of gas flow of 10, 15 and 30 cu. ft. per hour, while the results of tests with a rate of gas flow of 45 cu. ft. per hour were 5–10 per cent. low.

Method of Distillation and Baumé Gravity Determination.—The distillation apparatus and the method for the determination of the Baumé gravity of the distillate have been described by Oberfell.[1] The distillations were made after adding 500 c.c. of "straw oil" (a petroleum distillate having a gravity of about 30°Bé.) to the charcoal in the distilling flask. Distillation tests were also made in which 400 c.c. of glycerin were added to the charcoal contained in a 1,000-c.c. "Pyrex" flask. The distillations were continued until the thermometer placed in the vapors showed a temperature of 250°C. Very little gasoline was obtained after a temperature of 120°C. had been reached.

Several methods of distillation were investigated by Oberfell. Direct heating with the flame impinging against the flask was unsatisfactory for two reasons: first, it was difficult to drive off the vapors from the absorbing medium; and second, in tests of natural gas a large quantity of the less readily condensable vapors was absorbed and caused large distillation losses by carrying over some of the more readily condensable vapors. Oil-baths and sand-baths were found to be unsatisfactory because the temperature was not sufficient to expel the absorbed vapors. Steam distillation introduced difficulties in the Baumé gravity determinations and gave low yields.

The most satisfactory methods so far tried are distilling in the presence of straw oil or of glycerin. The advantage of using glycerin is twofold: first, the charcoal can be regenerated easily by washing with water; and second, gasoline is not appreciably soluble in glycerin or glycerin-water solutions. If the distillate is contaminated with oil, an error will be introduced in calculating the yields of gasoline, since separation of oil and gasoline is not practical with such a small volume of gasoline as is obtained in a test. Distillation tests in which glycerin was used have shown that when 10 c.c. of kerosene (45°Bé.) were added to the charcoal, 87 per cent. was recovered; and that when 25 c.c. of gasoline (65°Bé.) were added to the charcoal, 94 per cent. was recovered. The temperature of the condenser bath in each case was 0°C. The loss was due to the failure to remove the hydrocarbons completely from the charcoal and to the fact that some of the hydrocarbons could

[1] *J. Ind. Eng. Chem.*, **10** (1918), 211.

not be condensed since a certain amount was required to saturate the fixed gases (air, carbon dioxide, nitrogen, etc.) driven from the charcoal.

Extensive tests have not been carried out to determine how many times charcoal can be regenerated and thereby continue to be of value for absorption tests of this nature. This would depend, of course, upon several factors, chief of which is the quality of the charcoal. However, charcoal which had been regenerated three times gave satisfactory results. The method of regeneration is as follows: The glycerin is decanted off the charcoal. The charcoal then is placed on a Büchner funnel and washed with about 500 c.c. of hot water. The decanted glycerin may be used without further treatment or the wash water may be added to it and the combined solutions filtered and evaporated until all the water has been removed and the glycerin thus recovered and used again. The charcoal is left on the Büchner funnel and a stream of tap water is run through it for two to four hours. At the end of this period, the charcoal is placed in an oven and dried for several hours at about 140°C. (usually over night).

In making distillation tests, the condenser tube is surrounded by an ice-water bath or by a salt-water ice-bath. If desired, the vapors not condensed by an ice-water bath may be passed through a tube surrounded by a carbon dioxide—acetone mixture in a Dewar flask. The condensates thus obtained are combined and the Baumé gravity determined by means of a small hydrometer. The Baumé gravity of a distillate obtained by use of a salt-water ice-bath generally runs higher than 90°. The yield for different Baumé gravities may be determined by weathering the combined condensates obtained from duplicate tests. The above distillation methods offer the advantage of making a direct determination of the Baumé gravity of the gasoline. A modification of the method of distillation has been devised by P. M. Biddison and C. L. Veress, of the Ohio Fuel Supply Co. This modification consists in passing the uncondensed vapors through 25 c.c. of naphtha in a tall graduate. The condensate and the increase in volume of naphtha are taken into consideration in calculating the total yield of gasoline.

Applicability of the Method. (1) *Testing Efficiency of Plant Operation.*—The apparatus and method described have been used in testing natural gas and in checking the efficiency of the operation of gasoline plants.[1]

It has been found that the charcoal absorption method, when applied to residual gas from gasoline plants, gives results approximately 100 per cent. high, since the gasoline recovered by the method must be weathered about 50 per cent. before the Baumé gravity is as low as that which will in plant practice pass as usable product. Therefore, one simply divides these results by two to get the correct amount of

[1] See Bureau of Mines *Tech. Paper* **87.**

gasoline in the residual gas, or the gasoline content of the desired Baumé gravity may be obtained by weathering the combined condensate from duplicate tests.

(2) *Testing Casinghead Gas.*—Casinghead gases of about 70 oil wells of Roane County, W. Va., have been examined. These wells were tested for specific gravity, volume of gas and yield of gasoline. The results of a few of these tests, characteristic of the wells tested, are presented in this tabular form:

Well No.	Specific gravity of gas (air = 1)	Volume of gas, cu. ft. per 24 hrs.	Yield in gallons of 84°Bé. gasoline per M.
1	0.70	75,000	0.42
2	0.72	17,200	0.50
3	0.75	7,800	0.74
4	0.77	3,000	1.08
5	0.80	4,670	1.25
6	0.84	900	1.45
7	0.92	900	1.78

The results of the large number of tests made of the gases of this locality show that there is a fair relation between the specific gravity and the yield of gasoline. However, it is not to be inferred that these same relations hold for gases in all fields. Tests have shown that gases of different localities (and even gases of one locality) may show different specific gravity-yield curves.

Distillation of Gasoline Recovered by Charcoal Absorption Method.—The proportions of the gasoline extracted by the charcoal absorption method depend to a great extent upon the absorption value of the charcoal and the amount of gasoline absorbed per unit volume of absorbent.

Standard distillation tests were made of gasoline recovered by the charcoal absorption method in which the glycerin distillation method and the oil distillation method were employed. The gasoline obtained by the oil distillation method represents the average product of about 50 different tests. The gasoline obtained by the glycerin distillation method represents the average product of about 70 different tests. No appreciable difference has been found in results of tests which can be attributed to the method of removing the gasoline from the charcoal. However, preference is given to the glycerin distillation method for the reasons stated previously.

Table for Converting Gasoline Yield of Known Baumé Gravity to Yield of Another Baumé Gravity Desired as a Standard.—Natural gas which contains gasoline vapors in most cases contains a greater amount of the hydrocarbons of lower carbon content than of higher carbon con-

RESULTS OF TESTS OF GAS AT GASOLINE PLANTS

Source of gas	Charcoal absorption method		Oil absorption method		Plant production absorption	Remarks
	Plant 1; inlet	Plant 1; outlet	Plant 1; inlet	Plant 1; outlet	Plant 1; yield	
Gasoline recovered, Bé. 60°F./60°F...	90.2	92.0	90.4	91.0	88.6	It is not possible to recover and dispose of as salable product by plant practice more than 50 per cent. of this yield.
Gasoline yield, gallons per M. cu. ft. gas...	1.76	0.80	1.65	0.40	1.55	

Source of gas	Plant 2; inlet	Plant 2; outlet	Plant 2; inlet	Plant 2; outlet	Absorption plant 2; yield
Gasoline recovered, Bé. 60°F./60°F....	85.0	90.0	....	....	87.0
Gasoline yield, gallons per M. cu. ft. gas...	2.87	0.22	....	....	2.70

Source of gas	Plant 3; inlet	Plant 3; outlet	Plant 3; inlet	Plant 3; outlet	Plant 3; yield
Gasoline recovered, Bé. 60°F./60°F....	87.9	89.0	87.5	82.6	82.0 absorption
Gasoline yield, gallons per M. cu. ft. gas...	2.77	0.96	2.09	0.43	2.00 compression

Source of gas	Plant 4; inlet	Plant 4; outlet			Plant 4; yield
Gasoline recovered, Bé. 60°F./60°F....	85.0	....	....	....	81.0
Gasoline, gallons per M. cu. ft. of gas....	1.83	1.13	....	....	1.41

tent. In most cases it has been found that the proportion of propane is in excess of the percentage of butane, the percentage of butane in excess of that of the pentane, and so on. The greater the amount of the hydrocarbons of lower carbon content present in the gasoline of plant production, the greater the yield. The amounts of these hydrocarbons of lower carbon content naturally are limited by the properties of the gasoline offered for sale, since the product must meet certain specifications in order to pass as a salable product.

It has been customary for operators to consider the Baumé gravity

("gravity") of the gasoline an index to its properties, and accordingly to express the results of tests of gas in terms of yield of a certain Baumé gravity which has been found in plant practice to represent the maximum amount of salable product which can be recovered from a given volume of the gas.

In testing natural gas by the charcoal absorption method, it has been found, as one would expect, that the properties of the gasoline extracted depend to a great extent upon the absorption value of the charcoal and upon the amount of gasoline absorbed per unit volume of absorbent. When the amount of gasoline recovered per unit volume of absorbent is small, there occurs relatively large losses during distillation, with a subsequent lowering of the Baumé gravity of the distillate. Again, the Baumé gravity of the material will be low if the amount of gasoline absorbed per unit volume of absorbent is very large, since there is a tendency for the more volatile vapors to pass through the layer of charcoal without being absorbed and a tendency for those already absorbed to be driven out by the absorption of the less volatile vapors. The charcoal, as has been shown by different investigators, absorbs, before reaching saturation, a greater amount of vapors of the higher molecular weight than it does of the lower molecular weight.

The same general statements are true concerning the oil absorption method, and emphasize the necessity of making Baumé gravity determinations if one desires to express the results of tests in terms of yields of a definite Baumé gravity which has been accepted as the standard for maximum production.

The following table, which is intended for use in converting yield of known Baumé gravity to a yield of a different Baumé gravity, was prepared from data obtained in experiments on weathering losses of five different samples of gasoline from five different gasoline plants. To convert yield of a certain observed Baumé gravity to yield of another Baumé gravity accepted as a standard, multiply by the corresponding factor in the column under the desired standard Baumé gravity. *E.g.*, a distillate was obtained which had a Baumé gravity of 84° and the yield calculated from the volume of distillate was 2.00 gallons per 1,000 cubic feet of gas: if the gasoline extracted from this gas in plant operation has a Baumé gravity of 88°, the yield of 88° Bé. gasoline should be $2.00 \times$ factor or $2.00 \times 1.280 = 2.56$ gallons per 1,000 cubic feet of gas. Any correction factor would be subject to the composition of the gasoline extracted from a given gas. However, in several instances in tests of gas in the Mid-Continent field, yields obtained for different Baumé gravities have been compared by weathering the distillates from tests and close agreement has been observed with yields calculated (by use of factors) for the same Baumé gravities. It has been found, as one would expect, that the result obtained by calculating an 84°Bé.

yield to an 88°Bé. yield is somewhat lower than actually is obtained in tests of the gas from high pressure gas wells and in tests of the outlet gas of gasoline plants—the percentage difference in each case being small if correction factors are not used over a greater range than 5°Bé.

TABLE FOR CONVERTING GASOLINE YIELD OF KNOWN BAUMÉ GRAVITY TO YIELD OF ANOTHER BAUMÉ GRAVITY DESIRED AS THE STANDARD

Observed Baumé	Desired Baumé 89°	88°	87°	86°	85°	84°	83°
92	0.813	0.775	0.732	0.689	0.646	0.606	0.570
91	0.908	0.865	0.817	0.769	0.721	0.676	0.636
90	0.950	0.905	0.855	0.805	0.754	0.707	0.666
89	1.000	0.953	0.900	0.047	0.794	0.745	0.701
88	1.050	1.000	0.945	0.889	0.833	0.782	0.736
87	1.112	1.059	1.000	0.943	0.883	0.828	0.779
86	1.181	1.125	1.062	1.000	0.938	0.879	0.827
85	1.260	1.200	1.133	1.067	1.000	0.938	0.883
84	1.344	1.280	1.209	1.137	1.067	1.000	0.942
83	1.426	1.360	1.284	1.209	1.133	1.062	1.000
82	1.522	1.450	1.369	1.289	1.208	1.133	1.066
81	1.617	1.540	1.454	1.369	1.283	1.203	1.132
80	1.716	1.635	1.544	1.453	1.362	1.277	1.203
79	1.821	1.735	1.638	1.542	1.446	1.356	1.276
78	1.821	1.830	1.728	1.626	1.525	1.430	1.346
77	2.021	1.925	1.818	1.711	1.604	1.504	1.416
76	2.131	2.030	1.916	1.804	1.692	1.586	1.494
75	2.247	2.140	2.020	1.902	1.783	1.672	1.573
74	2.373	2.260	2.133	2.009	1.883	1.766	1.661
73	2.499	2.380	2.248	2.116	1.983	1.860	1.750
72	2.625	2.500	2.360	2.222	2.082	1.953	1.838

The principle of this method for testing natural gas for gasoline content consists in absorbing the vapors in a solid absorbing medium, such as charcoal, and subsequently recovering the gasoline by distillation. The method possesses several distinct advantages:

1. The method is accurate and rapid, the time consumed in absorbing the gasoline vapors from casinghead gas in a test by the charcoal absorption method being about 10 minutes.

2. The apparatus is simple to construct, easy to operate, and is readily portable, a portable outfit containing equipment necessary for 13 tests weighing 22 pounds.

3. The method gives information both as to yield and Baumé gravity of gasoline, and is applicable to both lean and rich natural gas.

4. Pressure is not required for absorption of the gasoline vapors.

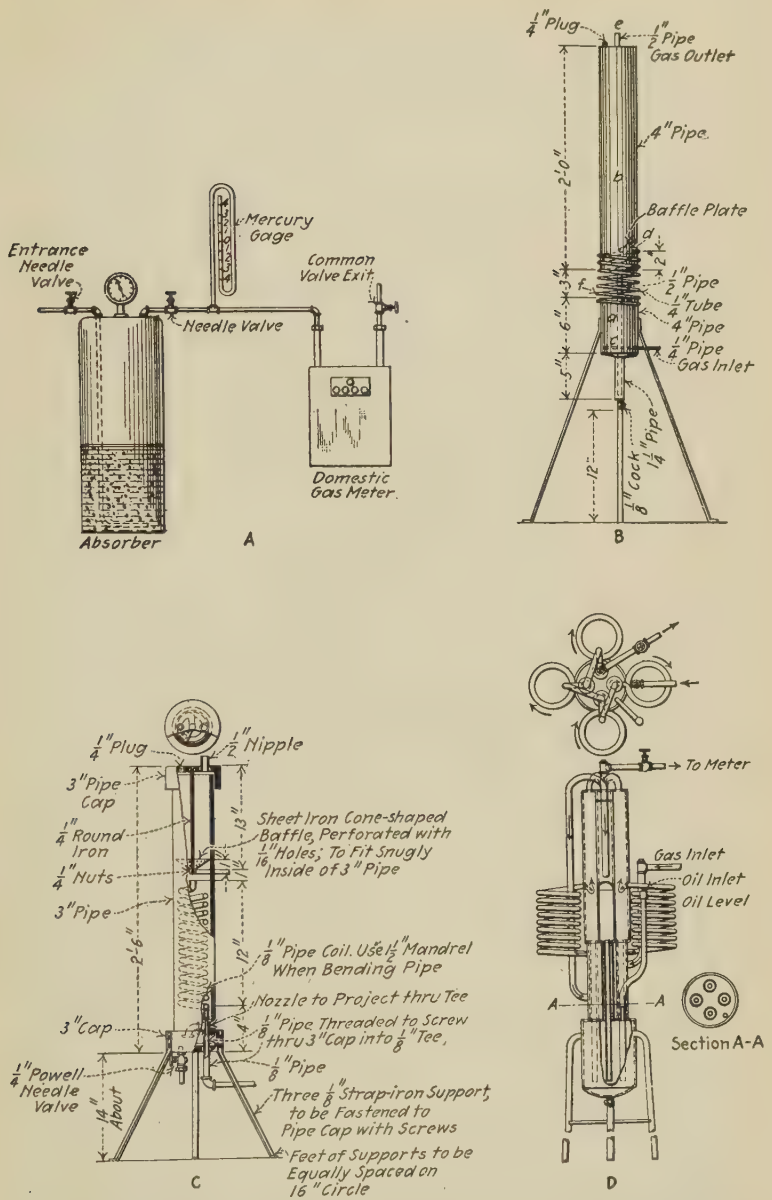


FIG. 101.—Various forms of apparatus for making absorption tests. *A* is the original autoclave absorber, which is convenient for rough reconnaissance work in the field. *B* is a single-coil absorption tester. *C* is a test-absorber of the induction type. *D* is the Biddison multiple-coil tester.

(*c*) Other Methods for Testing Natural Gas for Gasoline Content.—In the laboratory, the solubility of natural gas in various solvents often is used as a means of estimating the gasoline content of the gas. In making this test,[1] 35 c.c. of the absorbing medium are placed in a gas pipette over water or mercury and 100 c.c. of the gas are passed in from a burette. The pipette then is shaken for about 3 minutes, and the gas is returned to the burette and the volume read. Finally, the gas is passed into the pipette again, the pipette shaken, the gas returned, and the volume read. Pure methane is soluble in claroline oil

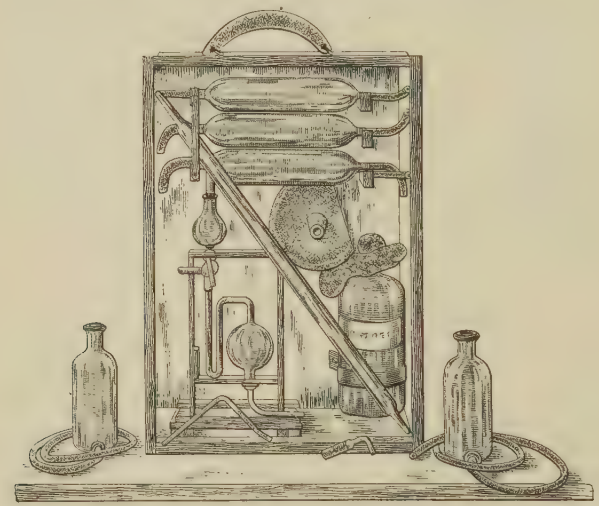


Fig. 102.—Apparatus for determining the gasoline content of casinghead gas. This portable outfit has been designed especially for the use of the non-technical man; it consists of a burette, pipette, gas collecting tubes, and one quart of Russian white oil.

to the extent of 11 per cent., while pure ethane is soluble to the extent of 68.5 per cent. Pittsburgh natural gas is soluble in claroline oil to the extent of 15.5 per cent., while in mineral seal oil it is soluble to the extent of 16.5 per cent.

Another apparatus for the determination of gasoline in natural gas, is that devised by Smith, Emery Co., of Los Angeles, Cal., and described by Dykema.[2] The gas from the well first is led through a meter and then through a coil, at a pressure of 12 in. of water. The coil is of ¾-in. copper, 15 ft. in length, and is

[1] Bureau of Mines *Tech. Paper* **87.**

[2] Bureau of Mines *Bulletin* **151.**

cooled by acetone mixed with carbon dioxide which has been added in the solid form. In this way the temperature of the acetone surrounding the coil may be held at the temperature of 70°F. below zero. The Newton absorption tester, a simple field apparatus, is described in Westcott's "Hand Book of Natural Gas" (3rd ed., 663).

An apparatus for the direct determination of gasoline in natural gas, using a small sample of the gas, has been described by Oberfell.[1]

The Davis differential pressure method, described in *J. Ind. Eng. Chem.*, **10** (1918), 709, 712 and 718, now is under investigation as to its applicability for the determination of the gasoline content of natural gas.

6. The Evaluation of Natural Gas for Carbon Black.[2]—The testing of natural gas for carbon black yield, preliminary to erecting plants, is very important. The variations in the actual amounts of carbon black obtained by the same process when using different gases are shown in the following table. The quantity recovered very closely follows the percentage of ethane, heating value, and amount of elementary carbon calculated from the hydrocarbons determined by analysis. The gas from Louisiana has the lowest percentage of nitrogen and the highest percentage of hydrocarbons, namely, 97.56, yet, when burned at a factory, gives the smallest yield of carbon black. This is due to the large proportion of methane that contains 33.5 pounds of carbon per 1,000 cu. ft., whereas the same quantity of ethane contains 67 pounds of carbon. The two gases from West Virginia have very similar compositions and give approximately the same yields of carbon black. The gas supplied to the plant in Wyoming has the largest calorific value and carbon content, and considerably more ethane than any of the other gases, and gives the greatest yield of carbon black. With the richer gases, not only the actual quantity of carbon obtained is larger, but the percentage recovery also increases. An examination of the chemical analysis of a gas is probably as trustworthy an indication of the available

[1] *J. Ind. Eng. Chem.*, **10** (1918), 211.

[2] Contributed by Roy O. Neal, Chemical Engineer, U. S. Bureau of Mines. For a select bibliography of lampblack and carbon black, see McClelland's "Lampblack," Carnegie Library, Pittsburgh, Pa., **1919.** On the testing of carbon black for quality, see Perrott and Thiessen, *J. Ind. Eng. Chem.*, **12** (1920), 324. These authors also propose tentative specifications for carbon black for various purposes.

carbon black as the result obtained with a small portable apparatus wherein the gas is burned and the deposited carbon black is collected and weighed.

CARBON CONTENT AND QUANTITY OF CARBON BLACK RECOVERED BY CHANNEL PROCESS[1]

	Louisiana, per cent. (*a*)	West Virginia, per cent. (*b*)	West Virginia, per cent. (*c*)	Wyoming, per cent. (*d*)
Methane	94.12	70.75	65.23	46.45
Ethane	3.44	24.14	30.07	43.10
Carbon dioxide	0.50	0.28	1.56	0.96
Nitrogen	1.94	4.83	3.14	9.49
Heating value[2]	962	1086	1134	1176
Pounds of carbon per 1,000 cu. ft. of gas[3]	33.8	39.9	42.3	44.3
Reported obtained as carbon black, lb. per M	0.80	1.00	1.10	1.40
Per cent. recovery	2.4	2.5	2.6	3.1

Neal has designed an apparatus for determining the quantity of carbon black which one might expect to obtain from an unknown natural gas. Details of the apparatus are shown in Fig. 103. It is made of 16-gage sheet iron and is held together with $\frac{1}{8}$-in. rivets. The carbon black is deposited upon a plate, 8 in. $\times$ 10-$\frac{3}{4}$ in. $\times$ $\frac{1}{4}$ in., and is removed by a scraper, 1 in. $\times$ 8 in. $\times$ $\frac{1}{16}$ in., which is operated by hand. The draft is controlled by eight 1-in. ventilating holes that are provided with covers which are pivoted above the holes and by a revolving damper located at the top of the hood. The pipe carrying the lava tips can be lowered below the depositing plate and held at any distance by attaching a plug in the stopper holes below the supports. The hopper carries a sliding door for removing the carbon black. Lava tips capable of burning about 4 cu. ft. of gas per hour are used. The hood, hopper and legs are detachable and fit into the central portion of the device to facilitate transporting in the field.

[1] Chemical analyses of gases were made by Donald B. Dow, Junior Chemist of the Bartlesville Station, U. S. Bureau of Mines.

[2] Net heating value in B.t.u. per cu. ft. at 0°C. and 760 mm. pressure.

[3] Calculated from the carbon contents of methane and ethane.

The apparatus ready for transporting for field use is shown in Fig. 104.

The apparatus can be used in the field by connecting it directly to the supply of gas and using suitable wind breaks if assembled in the field, or it can be used in the laboratory by obtaining a sample of gas under pressure in a steel cylinder.

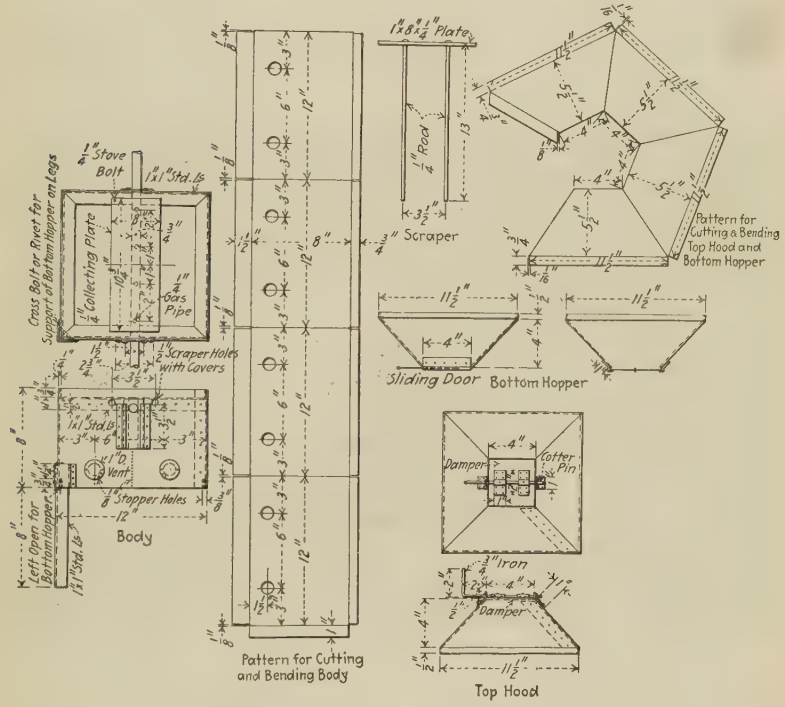


FIG. 103.—Detail drawing of the Neal apparatus for estimating the yield of carbon black from natural gas.

The source of supply of gas is connected with a meter registering at least as small a quantity as 0.1 cu. ft. and on this line should be a ¼-in. needle-valve in order to limit the flow of gas through the apparatus. Between the meter and the apparatus where the gas is burned, is attached a U-tube for determining the pressure of the gas. The top of the lava tips should be about 2½ in. below the depositing plate and a gas pressure of from ½ to 1 in. of water should be maintained. But these factors will be changed when examining different gases and in the preliminary

tests changes should be made until the largest recovery is obtained. A test should last at least an hour, during which time 15 or 20 cu. ft. of gas should be burned. It is possible to make a test without using the meter in the field by previously calibrating the apparatus against a meter in which different pressures on the manometer tube are observed and the time recorded. This will give fairly accurate data on the volumes, provided, of course, the lava tips are not broken or do not become plugged or partially obstructed by foreign matter after the apparatus has been standardized. During the test the carbon black should be removed

FIG. 104.—The apparatus of Neal for ascertaining the carbon black yield of natural gas. The illustration on the right shows the apparatus detached and ready for transportation.

from the collecting plate by the scraper every ten minutes, and at the end of the test the carbon which is deposited on the sides of the apparatus and plate is removed by use of a small brush. This accumulated carbon black then is weighed. From the data on the volume of gas burned, the quantity of carbon black collected, and the pressure of the gas, the quantity of carbon black which one might expect to obtain in plant operation from a given gas can be calculated. The apparatus assembled is shown in Fig. 104.

In both operations only comparative results are obtained. The portable apparatus will give results approximately parallel-

ing those procured in commercial plants using the channel system; but if some other process is used, it is necessary to standardize the apparatus against a plant where conditions will be similar to those where the gas in question is examined.

7. The Measurement of Natural Gas.[1] (*a*) CALIBRATION OF METERS. *Standard Meter Provers.*—The meter prover is the standard instrument by which the proof of a meter is ascertained. All meter provers should be calibrated by means of a standardized cubic-foot bottle.

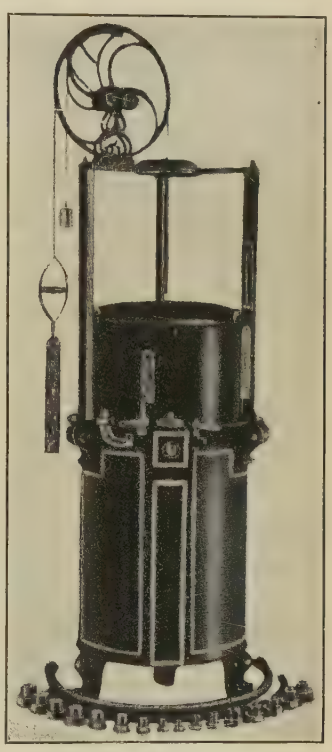


FIG. 105.—Standard meter prover.

The meter prover consists of a tank containing water in which is suspended a bell or holder having a supporting chain going over a large balance wheel. At the end of this chain is a weight holder with weights to give the desired gas pressure inside the bell. To the axis of the balance wheel is attached an involute with a counterpoise weight, the purpose of which is to maintain a uniform pressure at all points of travel of the bell. The wheel, chain, involute and weights are supported by a frame-work consisting of three columns and a triangular bridge across the top of the columns. The bases of the columns are screwed to sockets in the top of the tank. The bell is guided by three rollers at the bottom and three at the top of the bell. On the front of the bell is a scale properly graduated in cubic feet and fractions thereof, by means of which

[1] Contributed by H. R. Auerswald, Meter Expert, The Gypsy Oil Company. The scope of this treatise does not allow of a full consideration of the subject of the measurement of natural gas. Consequently, an account of the principles involved in such measuring devices as the Thomas electric meter, proportional meters, orifice meter, Venturi meter, and orifice provers ("flow meters") has been omitted.

is determined the exact amount of gas or air passed through a meter during its test.

On the front of the body is a channel having at its top a valve and two cocks, right and left hand. A hose is attached to either one of the cocks, as desired. On the outer end of this hose is a coupling for attaching to the meters to be tested. The connection to the meters is made by using intermediate reducers or increasers called inlet connections, except for one size which the hose coupling will fit, usually the ten-light meter. Two thermometers are provided for each prover, one to give the temperature of the water and the other that of the air. A 6-in. siphon gage also is furnished to give the pressure under which the prover is being operated.

These provers usually are constructed entirely of galvanized iron, either japanned or painted. They also are made with brass tanks, or bodies, and japanned and polished copper bells. The latter form is preferred by many engineers on account of its great durability. The regular sizes are 2-foot, 5-foot, 10-foot, and 20-foot capacity.

The Cubic-Foot Bottle.—This instrument is the basis of all gas measurement. In fact, the correctness of any gas measuring device, in its final analysis, is determined by the cubic-foot bottle, which is standardized by the Bureau of Standards at Washington, D. C., and hence possesses an accuracy which is beyond dispute.

The principle upon which the operation of the cubic-foot bottle is based, is very simple indeed; namely, a volume of 1 cu. ft. of gas is displaced by a volume of 1 cu. ft. of water. The mechanical detail required to do that conveniently and accurately is, however, more complex. As shown in Fig. 106, there is a cabinet containing and supporting the cubic-foot bottle, its system of piping and its tanks. The bottle, as the copper receptacle in the center of the cabinet is called, has a capacity of 1 cu. ft. At its top and bottom are gage glasses with pointers. When the water rises from the bottom pointer to the top one, 1 cu. ft. of gas has been delivered.

The operation is as follows: The lower tank is filled with water and this water is pumped to the upper tank. All temperatures of water, room and instrument to be tested are equalized. Now close all cocks, open the vent above the bottle, and open the cocks that admit the water from the top tank to the bottom of

the bottle and allow the water to come to the pointer on the lower gage glass. At this instant close the lower cock. Then close the vent and open the cocks in the line of piping leading to the article being tested. Next reopen the cock admitting water to the bottle. Allow the water to fill the bottle and to come to the pointer on the upper gage glass. Close the water cock and the piping cock. Then 1 cu. ft. of air has been delivered. Open the vent and the cock admitting water to the lower tank and allow to drain out of the bottle. Next pump the water from the lower tank to the upper. Then repeat the method of procedure of operation of the bottle, if successive cubic feet are desired. Extreme care must be exercised always to have the temperatures exact and unvarying. In some instances the operation must be conducted in a room in which the air is saturated with water vapor.

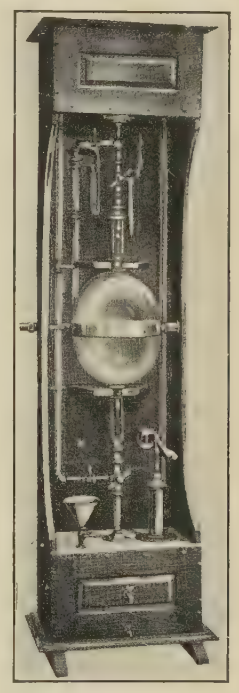


FIG. 106.—Cubic-foot bottle. For calibrating laboratory meters, a bottle of $\frac{1}{10}$-cu. ft. capacity generally is used.

The Correction of Erratic Meters (by F. H. Oliphant).—A fast meter is one which registers too many cu. ft. and a slow meter is one which registers too few cu. ft., as compared with a prover which measures the correct number of cu. ft. and which is the standard to which all meters are compared. The multipliers given in the following table are all less than one for fast meters and greater than one for slow meters.

A meter on which the dial shows 10.5 cu. ft. when the prover shows 10 cu. ft. is called 5 per cent. fast and must be multiplied by 0.952 to reduce the quantity to standard. A meter on which the dial shows 9.5 cu. ft. when the prover shows 10 cu. ft. is termed 5 per cent. slow and must be multiplied by 1.053 to bring it up to the standard.

Because the dial of many meters cannot be read as accurately as the scale on the prover, it is preferred in some cases to pass the air or gas through the meter and prover until the meter registers 10 cu. ft., then shutting off and reading the prover scale. For

this purpose a second table is introduced which, however, is consistent with the first. This method simplifies the computation for the multiplier, which shows directly from the prover scale, being one-tenth the value of the prover scale reading.

The correction factor or multiplier to correct erratic meters is determined by the following formula:

$$\text{Multiplier} = \frac{\text{Prover reading}}{\text{Meter reading}}.$$

Example: Say the reading of a meter is 10.0, while the prover reads 12.5; then the multiplier is $\frac{12.5}{10} = 1.25$. Or, say the reading of the meter is 10 when the prover reads 8; then $\frac{8}{10} = 0.8$ is the multiplier.

The formula for determining the percentage that a meter is fast is as follows:

$$\frac{(\text{Meter reading} - \text{Prover reading}) \times 100}{\text{Prover reading}} = \text{percentage error fast.}$$

Example: Say a meter registers 10 cu. ft. while the prover shows 8; then $\frac{(10 - 8) \times 100}{8} = \frac{200}{8} = 25$ per cent. error fast.

The formula for determining the percentage error of a slow meter is as follows:

$$\frac{(\text{Prover reading} - \text{Meter reading}) \times 100}{\text{Prover reading}} = \text{percentage error slow.}$$

Example: Say a slow meter registering 10 showed 12.5 cu. ft. on the prover; then $\frac{(12.5 - 10) \times 100}{12.5} = \frac{250}{12.5} = 20$ per cent. error slow.

The multipliers for slow and fast meters are determined by the following formulas: Multipliers for meters that are slow, $\frac{100}{100 - \text{per cent. slow}}$. Multipliers for meters that are fast, $\frac{100}{100 + \text{per cent. fast}}$.

Example: Suppose a meter is said to be 20 per cent. slow. In this case, the multiplier $= \frac{100}{100 - 20} = \frac{100}{80} = 1.25$. On the other hand, suppose a meter is reported 25 per cent. fast. Here the multiplier $= \frac{100}{100 + 25} = \frac{100}{125} = 0.80$.

TABLE GIVING MULTIPLIERS FOR CORRECTION OF ERRATIC REGISTER OF METERS, SLOW AND FAST

Meter reading, cu. ft.	Slow meters			Fast meters		
	Prover reading, cu. ft.	Percentage of variation (prover being Standard)	Multipliers to correct slow meters	Prover reading, cu. ft.	Percentage of variation (prover being Standard)	Multipliers to correct fast meters
10	13.7	27.00	1.37	10.0	0.00	1.00
10	13.6	26.47	1.36	9.9	1.01	0.99
10	13.5	25.93	1.35	9.8	2.04	0.98
10	13.4	25.37	1.34	9.7	3.09	0.97
10	13.3	24.81	1.33	9.6	4.17	0.96
10	13.2	24.24	1.32	9.5	5.26	0.95
10	13.1	23.66	1.31	9.4	6.38	0.94
10	13.0	23.08	1.30	9.3	7.53	0.93
10	12.9	22.48	1.29	9.2	8.70	0.92
10	12.8	21.88	1.28	9.1	9.89	0.91
10	12.7	21.26	1.27	9.0	11.11	0.90
10	12.6	20.63	1.26	8.9	12.36	0.89
10	12.5	20.00	1.25	8.8	13.63	0.88
10	12.4	19.35	1.24	8.7	14.94	0.87
10	12.3	18.70	1.23	8.6	16.28	0.86
10	12.2	18.03	1.22	8.5	17.65	0.85
10	12.1	17.35	1.21	8.4	19.05	0.84
10	12.0	16.67	1.20	8.3	20.48	0.83
10	11.9	15.97	1.19	8.2	21.95	0.82
10	11.8	15.26	1.18	8.1	23.46	0.81
10	11.7	14.53	1.17	8.0	25.00	0.80
10	11.6	13.80	1.16	7.9	26.58	0.79
10	11.5	13.04	1.15	7.8	28.20	0.78
10	11.4	12.28	1.14	7.7	29.87	0.77
10	11.3	11.50	1.13	7.6	31.58	0.76
10	11.2	10.71	1.12	7.5	33.33	0.75
10	11.1	9.91	1.11	7.4	35.13	0.74
10	11.0	9.09	1.10	7.3	37.00	0.73
10	10.9	8.26	1.09	7.2	38.88	0.72
10	10.8	7.41	1.08	7.1	40.84	0.71
10	10.7	6.54	1.07	7.0	42.86	0.70
10	10.6	5.66	1.06	6.9	44.93	0.69
10	10.5	4.76	1.05	6.8	47.06	0.68
10	10.4	3.85	1.04	6.7	49.26	0.67
10	10.3	2.91	1.03	6.6	51.51	0.66
10	10.2	1.96	1.02	6.5	53.85	0.65
10	10.1	0.99	1.01	6.4	56.25	0.64
10	10.0	0.00	1.00	6.3	58.73	0.63

In other words, to correct a meter reading, divide it by 1 plus the per cent. expressed as a decimal fraction that the meter is fast, or by 1 minus the per cent. expressed as a decimal fraction that the meter is slow; or in each case by multiplying by the reciprocals of these quantities.

The Use of the Above Table.—If 10 cu. ft. in a meter showed only 7.5 cu. ft. in the prover, the meter is 33.33 per cent. fast. In the table opposite 7.5, multiplier 0.75 is found. Say a meter in use recorded 42,250 cu. ft. when disconnected and was found to be 33.33 per cent. fast, then $42,250 \times 0.75 = 31,687.5$ cu. ft., which is the corrected quantity. On the other hand, if a meter recording 10 cu. ft. gave 11.5 cu. ft. in the prover, the meter is 13.04 per cent. slow and in the table opposite 11.5 cu. ft. a multiplier of 1.15 is recorded. If the meter registered 42,250 cu. ft. when disconnected, then $42,250 \times 1.15 = 48,587.5$ cu. ft. is the correct quantity. It will be observed that the multiplier for correcting erratic meters is the quantity recorded by the prover with the decimal point moved one figure to the left. Then, if the prover shows 7.5 cu. ft. and the meter 10 cu. ft., the correcting multiplier is 0.75. On the other hand, if the prover shows 11.5 cu. ft. and the meter 10, the correcting multiplier is 1.15. If the prover should show 10.125 and the meter 10 cu. ft., the multiplier will be 1.0125. It is much more direct to use the multiplier than to reduce the percentage, to obtain the corrected quantity from an erratic meter.

If it is desired to get the error in the gas bill, instead of the error in the meter, when testing the meter, it can be obtained direct from the prover and meter readings. For instance, suppose 10 cu. ft. in a meter showed only 7.5 cu. ft. in the prover. The gas bill then would be $\frac{10 - 7.5}{10} = 0.25$, or 25 per cent. too much, and the same multiplier should be used as in correcting the meter reading. If the meter in use showed 12,500 cu. ft., the actual amount of gas passed was $12,500 \times 0.75$ or $12,500 - (12,500 \times 0.25) = 9,375$ cu. ft.

Suppose the meter was slow, and 10 cu. ft. on the meter showed 11.5 cu. ft. on the prover, as in the previous case, the gas bill would be $\frac{11.5 - 10}{10} = 0.15$, or 15 per cent. too small. If the meter in use showed 15,500 cu. ft., the actual amount of gas passed was $15,500 \times 1.15$ or $15,500 + (15,500 \times 0.15) = 17,825$ cu. ft.

If the meter tester makes his calculations in this manner, the gas accountant in the office can add or subtract the percentage as shown on the meter tester's report and thereby make a correction in the bill. This procedure often prevents the misunderstanding which is liable to arise if the correction applied to the gas bill is different than the percentage error of the meter. The error in the meter and the error in the gas bill are different, but the methods of correction are the same.

(*b*) Testing Natural Gas Wells for Volume. *Testing Natural Gas Wells with the Pitot Tube.*—When the producer drills into a natural gas sand, during the drilling the sudden rush of gas from the well sometimes throws the tools out of the hole and clear of the derrick. The well then is closed-in and as soon as possible a test is made to determine the capacity or open-flow volume of gas from the well. By open-flow volume is meant the number of cu. ft. of atmospheric gas which the well will produce daily when wide open to the air.

The results of the test afford useful information for the guidance of the owner or a prospective buyer of the gas from the well. It forms a basis for figuring how much gas may be taken from the well monthly if the rock and line pressures are such that the well will feed into the line a fair percentage of its open-flow volume. In some States it is permissible to take from a well 25 per cent. of its over-flow volume. The results of the test, however, cannot be used for computing how much gas the well will feed into a line which has a certain pressure on it. This amount can be obtained only by holding a pressure equal to the line pressure on the well and allowing enough gas to escape to just maintain that pressure at the top of the well. The escaping gas can be measured and the volume of gas which the well will produce against that line pressure is ascertained thereby. The measurement of the escaping gas can be accomplished in the same manner as the Pitot tube test for open-flow, which is described *infra*.

The Pitot tube consists of a piece of ¼-in. pipe bent at right angles in the form of a Z. To one end of the pipe is connected a U-gage or indicating pressure-gage. For small volume wells a U-gage filled with water is best; for larger volume wells mercury should be used, and for very large volume wells an indicating pressure-gage should be employed. The well should be opened and allowed to flow until the flow has settled to a regular volume.

This can be ascertained by holding the Pitot tube in the flow at 5-minute intervals until two or three like readings are obtained. Some wells will settle down in 15 minutes while others may take one hour or more.

To measure the flow of gas, the open end of the ¼-in. pipe is held in the stream of flowing gas so that the opening is at direct right angles to the flow, and ⅓ the way across the opening from which the gas is flowing. The pipe from which the gas is flowing should be straight, smooth, and of the same diameter for as many feet as it is inches in diameter. There should be no fittings, nor should the flow be measured from a collar or fitting of any kind. The highest velocity of flow is at the center of the pipe, and the lowest at the outside. The average velocity occurs one-third the distance across the pipe. The impact of the flowing gas against the open end of the Pitot tube pipe is transmitted to the surface of the liquid in the U-gage and the surface of the liquid is pushed down on one side of the gage and up on the other, until the static head of the liquid equals the velocity head of the gas. The reading is the difference in inches between the two surfaces of the liquid.

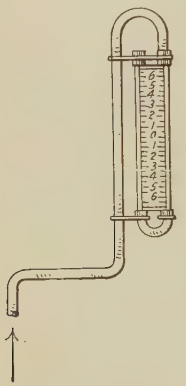


FIG. 107.—Pitot tube or siphon gage for measuring the volume of gas flowing from a natural gas well.

F. H. Oliphant has compiled the table presented below, from which the volume in cu. ft. per day can be obtained if the pressure on the U-tube is ascertained by test. The data are based on a 4-oz. pressure or 14.65 lbs. per sq. in., a gas temperature of 60°F., and a specific gravity of 0.6, compared with air. If the specific gravity of the gas is other than 0.6, the figures in the table should be multiplied by $\sqrt{\frac{0.6}{\text{true sp. gr.}}}$. If the temperature is above or below 60°F., 1 per cent. for each 10° should be deducted or added.

Pitot Tube Table for Testing of Gas Wells

Discharge of gas of 0.6 specific gravity from gas-well tubing of different sizes in twenty-four hours

Pressure			Discharge in cubic feet			
In in. of water	In in. of mercury	In lb. per sq. in.	1-in. tubing	2-in. tubing	3-in. tubing	4-in. tubing
0.10	.....	.....	11,880	47,520	106,920	190,080
0.20	.....	.....	17,136	68,544	154,224	274,176
0.30	.....	.....	20,568	82,272	185,112	329,088
0.40	.....	.....	23,520	94,080	211,680	376,320
0.50	.....	.....	26,544	106,176	238,896	424,704
0.60	.....	.....	29,112	116,448	262,008	465,792
0.70	.....	.....	31,440	125,760	282,960	503,040
0.8	.....	.....	33,624	134,496	302,616	537,984
0.9	.....	.....	35,640	142,560	320,760	570,240
1.0	.....	.....	37,320	149,280	335,880	597,120
1.25	.....	.....	41,712	166,848	375,408	667,392
1.5	.....	.....	45,960	183,840	413,640	735,360
1.75	0.12	.....	49,680	198,720	447,120	794,880
2.0	0.147	.....	53,136	212,544	478,224	850,176
2.5	0.184	.....	59,400	237,600	534,600	950,400
3.0	0.22	0.108	65,088	260,352	585,792	1,041,408
3.5	0.257	0.126	70,272	281,088	632,448	1,124,352
4.0	0.294	0.144	75,120	300,480	676,080	1,201,920
4.5	0.331	0.162	79,704	318,810	717,336	1,275,264
5.0	0.368	0.18	84,000	336,000	756,000	1,344,000
6.0	0.441	0.216	92,016	368,060	828,144	1,472,256
7.0	0.515	0.252	99,360	397,440	894,240	1,589,760
8.0	0.588	0.288	106,272	425,088	956,448	1,700,352
9.0	0.662	0.324	112,656	450,624	1,013,904	1,802,496
10.0	0.736	0.36	118,800	475,200	1,069,200	1,900,800
11.0	0.8	0.396	125,160	500,640	1,126,440	2,002,560
12.0	0.88	0.432	130,128	520,512	1,171,152	2,082,048
	1.02	0.5	138,960	555,840	1,250,640	2,223,360
	1.52	0.75	170,280	681,120	1,532,520	2,724,480
	2.03	1.00	196,680	786,720	1,770.120	3,146,880
	2.54	1.25	219,960	879,840	1,979,640	3,519,360
	3.05	1.5	240,720	962,880	2,166,480	3,851,520
	3.56	1.75	259,920	1,039,680	2,339,280	4,158,720

Pressure			Discharge in cubic feet			
In in. of water	In in. of mercury	In lb. per sq. in.	1-in. tubing	2-in. tubing	3-in. tubing	4-in. tubing
	4.07	2.00	272,640	1,090,560	2,453,760	4,362,240
	4.57	2.25	294,600	1,178,400	2,651,400	4,713,600
	5.08	2.50	310,800	1,243,200	2,797,200	4,972,800
	5.59	2.75	321,000	1,284,000	2,889,000	5,136,000
	6.10	3.00	340,200	1,360,800	3,061,800	5,443,200
	6.61	3.25	354,120	1,416,480	3,187,080	5,665,920
	7.11	3.50	367,680	1,470,720	3,309,120	5,882,880
	7.62	3.75	380,400	1,521,600	3,423,600	6,086,400
	8.13	4.00	392,880	1,571,520	3,535,920	6,286,080
	8.64	4.25	405,000	1,620,000	3,645,000	6,480,000
	9.15	4.50	416,640	1,666,560	3,749,760	6,666,240
	9.65	4.75	428,280	1,713,120	3,854,520	6,852,480
	10.16	5.00	439,920	1,759,680	3,959,280	7,038,720
	12.20	6.0	476,040	1,904,160	4,284,360	7,616,640
		7.0	517,320	2,069,280	4,655,880	8,277,120
		8.0	542,400	2,169,600	4,881,600	8,678,400
		9.0	569,640	2,278,650	5,126,760	9,114,240
		10.0	595,560	2,382,240	5,360,040	9,528,960
		11.0	621,960	2,487,840	5,597,640	9,951,360
		12.0	642,600	2,570,400	5,783,400	10,281,600
		13.0	664,680	2,658,720	5,982,120	10,634,880
		14.0	683,880	2,735,520	6,154,920	10,942,080
		15.0	703,080	2,812,320	6,327,720	11,249,280
		16.0	721,080	2,884,320	6,489,720	11,537,280
		17.0	738,120	2,952,480	6,643,080	11,809,920
		18.0	753,960	3,015,840	6,785,640	12,063,360
		20.0	785,520	3,142,080	7,069,680	12,568,320
		22.0	803,280	3,213,120	7,229,520	12,852,480
		25.0	854,880	3,419,520	7,693,920	13,678,080
		30.0	910,680	3,642,720	8,196,120	14,570,880
		35.0	960,960	3,843,840	8,648,640	15,375,360
		40.0	1,006,680	4,026,720	9,060,120	16,106,880
		45.0	1,046,520	4,186,080	9,418,680	16,744,320
		50.0	1,081,920	4,327,680	9,737,280	17,310,720
		60.0	1,137,120	4,548,480	10,234,080	18,193,920
		75.0	1,223,400	4,893,600	11,010,600	19,574,400
		90.0	1,304,400	5,217,600	11,739,600	20,870,400
		100.0	1,336,920	5,347,680	12,032,280	21,390,720

Multipliers for pipe diameters other than given in the above table.—For any different sized pipe, apply the multiplier to the figures given in the above table for "one-inch tubing."

1½-inch = 2.25	5-inch = 25.00	8-inch = 64.0
2½-inch = 6.25	5⅝-inch = 31.64	8¼-inch = 68.0
4¼ inch = 18.00	6-inch = 36.0	9-inch = 81.0
4⅝-inch = 21.39	6¼-inch = 39.0	10-inch = 100.0
	6⅝-inch = 43.9	12-inch = 144.0

The Orifice Well Tester.—The method just described for ascertaining the volume of gas flowing from a natural gas well when open to the air, is a very accurate procedure for measuring large wells, but it is not practical in measuring small volumes. To measure accurately small volumes from 1,000 to 400,000 cu. ft. of gas per day, such as usually are given off from the casingheads of oil wells (called "casinghead gas"), a different kind of apparatus must be used.

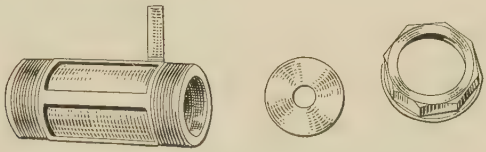


Fig. 108.—The orifice well tester.

An orifice prover could be employed for this purpose, but an orifice well tester is to be preferred. It consists of a short piece of 2-in. pipe with threads on both ends and with a short ⅛-in. pipe nipple in the side for connecting to a U-gage. The U-gage used is the same as shown in Fig. 107; it is connected to the short ⅛-in. nipple by a piece of ¼-in. gas hose and the pressure is taken by reading the difference in inches between the two surfaces of the liquid. For small volume wells, water can be used; for the large ones it is necessary to use mercury.

When taking the volume of a well, the left end (Fig. 108) of the orifice well tester is screwed into the casinghead. The gas should be allowed to blow out for such a time until its flow is regular or down to normal. When down to normal, the proper sized orifice is placed over the other end of the well tester and held in place by the cap. An orifice which is sufficiently large to have a small reading on the U-gage should be used, for a large pressure retards the flow from the well.

By the use of the table given in the *Appendix*, page 549, from the pressure reading on the U-gage and the specific gravity of the gas, the volume in cu. ft. per 24-hour day can be obtained. The table is supplied with well testers manufactured by the Metric Metal Works, Erie, Pa., and is correct only for orifices of that make. The orifices are square-edge and cut through plate $\frac{1}{8}$-in. thick. In case other manufacturers furnish an orifice of different design, a table determined by calibration should be supplied with the equipment. In case it is desired to measure a well which creates a pressure behind the orifice not given in the table, the volume can be ascertained by multiplying the volume found in the table, by the square root of the pressure actually obtained, divided by the square root of the pressure found in the table.

Example: Suppose a well has a volume such that it creates 7 in. of water on a $1\frac{1}{4}$-in. orifice. The specific gravity is 1.0. What is the daily volume?

From the table, under the specific gravity 1.0 and 5.5 in. of water, there is found 83,060. The volume of the well then is:

$$\frac{\sqrt{7}}{\sqrt{5.5}} \times 83{,}060 = 93{,}600 \text{ cu. ft. per day.}$$

This method of calculation can be explained by the formula for the theoretical velocity in feet per second of flow of any fluid through an orifice, which is—

$$V = \sqrt{2gh} = 8.02\sqrt{h},$$

in which h = the "head" or height of the fluid in feet required to produce the pressure of the fluid at the level of the orifice. (For gases the formula holds good only for small differences of pressure on the two sides of the orifice.) The quantity of flow in cu. ft. per second is equal to the product of this velocity by the area of the orifice, in square feet, multiplied by the coefficient of flow or "efficiency," which takes into account the contraction of the vein or flowing stream, the friction of the orifice, etc. This "efficiency" varies from 0.56 to 0.79 for circular orifices in thin plates.

From this formula it can be seen that the velocity through the orifice, and consequently the volume delivered, varies directly as the square root of the pressure behind the orifice plate registered by the U-gage.

From many oil wells the casinghead gas flows by "heads," *i.e.*, there is a strong flow of gas for a short time and then there is

practically no flow for an interval of time. In cases of this kind it is necessary to take the time between "heads" and determine how many flows there are per day. Then, by getting the average pressure on the orifice for the period of flow of each head, the volume of gas given off at each flow can be determined. In this way a fair estimate of the daily volume of the well can be ascertained.

APPENDIX

°C °F
50 120
45 110
40 100
35 90
30 80
25 70
20 60
15 65 150
10 50 60 140 110 230 160 320 210 410 260 500 310 590 360 680 410 770 460
5 40 55 105 130 155 220 205 310 255 400 305 490 355 580 405 670 455 760 860
0 32 50 122 100 212 150 302 200 392 250 482 300 572 350 662 400 752 450 842
100 210
95 200
90 190
85 180
75 170
70 160
120
115
105
130
145 300
140 290
135 280
130 270
125 260
175
165
155
195 390
190 380
185 370
180 360
175 350
170 340
165 330
215
205
245 480
240 470
235 460
230 450
225 440
220 430
215 420
265
255
295 570
290 560
285 550
280 540
275 530
270 520
315
305
345 660
340 650
335 640
330 630
325 620
320 610
365
355
395 750
390 740
385 730
380 720
375 710
370 700
415
405
445 840
440 830
435 820
430 810
425 800
420 790
465
455
495 930
490 920
485 910
480 900
475 890
470 880
515
505
545
535
525
565
555
595
585
575
615
605
645
635
625
665
655
695
685
675
715
705
745
735
725
765
755
795
785
775
815
805
845
835
825
865
855
895
885
875

Table Showing the Degrees Baumé at 60°F. of Oils Having, at the Designated Temperatures, the Observed Degrees Baumé Indicated. (From Bureau of Standards Circular 57.)

[This table shows the degrees Baumé at 60°F. of oils having, at the designated temperatures, the observed degrees Baumé indicated. For example, if the observed degrees Baumé are 20.0 at 78°F., the true degrees Baumé at 60°F. will be 19.0. Intermediate values not given in the table may be conveniently interpolated. For example, if the observed degrees Baumé are 20.4 at 78°F., the true degrees Baumé at 60°F. will be 19.4. The headings "Observed degrees Baumé" and "Observed temperature" signify the true indication of the hydrometer and the true temperature of the oil—that is, the observed readings corrected if necessary, for instrumental errors.]

Observed temperature in °F.	Observed degrees Baumé									
	17.0	18.0	19.0	20.0	21.0	22.0	23.0	24.0	25.0	26.0
	Corresponding degrees Baumé at 60°F.									
30	18.6	19.7	20.7	21.7	22.7	23.7	24.8	25.8	26.9	27.9
32	18.6	19.6	20.6	21.6	22.6	23.6	24.7	25.7	26.8	27.8
34	18.5	19.5	20.5	21.5	22.5	23.5	24.6	25.6	26.7	27.7
36	18.3	19.4	20.4	21.4	22.4	23.4	24.5	25.5	26.5	27.5
38	18.2	19.3	20.3	21.3	22.3	23.3	24.4	25.4	26.4	27.4
40	18.1	19.1	20.1	21.2	22.2	23.2	24.2	25.2	26.2	27.2
42	18.0	19.0	20.0	21.1	22.1	23.1	24.1	25.1	26.1	27.1
44	17.9	18.9	19.9	20.9	21.9	22.9	23.9	24.9	26.0	27.0
46	17.8	18.8	19.8	20.8	21.8	22.8	23.8	24.8	25.9	26.9
48	17.6	18.7	19.7	20.7	21.7	22.7	23.7	24.7	25.8	26.8
50	17.5	18.6	19.6	20.6	21.6	22.6	23.6	24.6	25.6	26.6
52	17.4	18.5	19.5	20.5	21.5	22.5	23.5	24.5	25.5	26.5
54	17.3	18.3	19.3	20.3	21.3	22.3	23.3	24.3	25.4	26.4
56	17.2	18.2	19.2	20.2	21.2	22.2	23.2	24.2	25.3	26.3
58	17.1	18.1	19.1	20.1	21.1	22.1	23.1	24.1	25.1	26.1
60	17.0	18.0	19.0	20.0	21.0	22.0	23.0	24.0	25.0	26.0
62	....	17.9	18.9	19.9	20.9	21.9	22.9	23.9	24.9	25.9
64	....	17.8	18.8	19.8	20.8	21.8	22.8	23.8	24.7	25.7
66	....	17.7	18.7	19.7	20.7	21.7	22.7	23.7	24.6	25.6
68	....	17.6	18.6	19.5	20.5	21.5	22.5	23.5	24.5	25.5
70	....	17.5	18.5	19.4	20.4	21.4	22.4	23.4	24.4	25.4
72	....	17.4	18.4	19.3	20.3	21.3	22.3	23.3	24.3	25.3
74	....	17.2	18.2	19.2	20.2	21.2	22.2	23.2	24.1	25.1
76	....	17.2	18.1	19.1	20.1	21.1	22.1	23.1	24.0	25.0
78	....	17.1	18.0	19.0	19.9	20.9	21.9	22.9	23.9	24.9
80	....	....	17.9	18.9	19.8	20.8	21.8	22.8	23.8	24.8
82	....	....	17.8	18.8	19.7	20.7	21.7	22.7	23.7	24.7
84	....	....	17.7	18.7	19.6	20.6	21.6	22.6	23.5	24.5
86	....	....	17.6	18.6	19.5	20.5	21.5	22.5	23.4	24.4
88	....	....	17.5	18.4	19.4	20.4	21.3	22.3	23.3	24.3
90	....	....	17.3	18.3	19.3	20.3	21.2	22.2	23.2	24.2
92	....	....	17.2	18.2	19.2	20.2	21.1	22.1	23.1	24.1
94	....	....	17.1	18.1	19.1	20.1	21.0	22.0	23.0	24.0
96	....	....	17.0	18.0	19.0	20.0	20.9	21.9	22.8	23.8
98	....	....	....	17.9	18.8	19.8	20.8	21.8	22.7	23.7
100	....	....	....	17.8	18.7	19.7	20.7	21.7	22.6	23.6
102	....	....	....	17.7	18.6	19.6	20.5	21.5	22.5	23.5
104	....	....	....	17.6	18.5	19.5	20.4	21.4	22.4	23.4
106	....	....	....	17.5	18.4	19.4	20.3	21.3	22.3	23.3
108	....	....	....	17.3	18.2	19.2	20.2	21.2	22.2	23.1
110	....	....	....	17.2	18.1	19.1	20.1	21.1	22.0	23.0
112	....	....	....	17.1	18.0	19.0	20.0	21.0	21.9	22.9
114	....	....	....	17.0	17.9	18.9	19.9	20.9	21.8	22.8
116	....	....	....	....	17.8	18.8	19.8	20.8	21.7	22.7
118	....	....	....	....	17.7	18.7	19.6	20.6	21.5	22.5
120	....	....	....	....	17.6	18.6	19.5	20.5	21.4	22.4

Observed temperature in °F.	Observed degrees Baumé									
	27.0	28.0	29.0	30.0	31.0	32.0	33.0	34.0	35.0	36.0
	Corresponding degrees Baumé at 60°F.									
30	29.0	30.0	31.0	32.0	33.1	34.1	35.2	36.2	37.3	38.3
32	28.8	29.8	30.9	31.9	33.0	34.0	35.0	36.0	37.1	38.1
34	28.7	29.7	30.8	31.8	32.8	33.8	34.8	35.8	36.9	38.0
36	28.5	29.5	30.6	31.6	32.7	33.7	34.7	35.7	36.8	37.8
38	28.4	29.4	30.5	31.5	32.5	33.5	34.5	35.5	36.6	37.7
40	28.3	29.3	30.4	31.4	32.4	33.4	34.4	35.4	36.5	37.5
42	28.2	29.2	30.2	31.2	32.2	33.2	34.3	35.3	36.3	37.3
44	28.1	29.1	30.1	31.1	32.1	33.1	34.2	35.2	36.2	37.2
46	27.9	28.9	29.9	30.9	31.9	32.9	34.0	35.0	36.1	37.1
48	27.8	28.8	29.8	30.8	31.8	32.8	33.9	34.9	35.9	36.9
50	27.6	28.6	29.7	30.7	31.7	32.7	33.7	34.7	35.7	36.7
52	27.5	28.5	29.6	30.6	31.6	32.6	33.6	34.6	35.6	36.6
54	27.4	28.4	29.4	30.4	31.4	32.4	33.4	34.4	35.4	36.4
56	27.3	28.3	29.3	30.3	31.3	32.3	33.3	34.3	35.3	36.3
58	27.1	28.1	29.1	30.1	31.1	32.1	33.1	34.1	35.1	36.1
60	27.0	28.0	29.0	30.0	31.0	32.0	33.0	34.0	35.0	36.0
62	26.9	27.9	28.9	29.9	30.9	31.9	32.9	33.9	34.9	35.9
64	26.7	27.7	28.7	29.7	30.7	31.7	32.7	33.7	34.7	35.7
66	26.6	27.6	28.6	29.6	30.6	31.6	32.6	33.6	34.6	35.6
68	26.5	27.5	28.4	29.4	30.4	31.4	32.4	33.4	34.4	35.4
70	26.4	27.4	28.3	29.3	30.3	31.3	32.2	33.2	34.2	35.2
72	26.3	27.3	28.2	29.2	30.2	31.2	32.1	33.1	34.1	35.1
74	26.1	27.1	28.1	29.1	30.1	31.1	32.0	33.0	33.9	34.9
76	26.0	27.0	27.9	28.9	29.9	30.9	31.8	32.8	33.8	34.8
78	25.8	26.8	27.8	28.8	29.8	30.8	31.7	32.7	33.6	34.6
80	25.7	26.7	27.7	28.7	29.7	30.7	31.6	32.6	33.5	34.5
82	25.6	26.6	27.6	28.6	29.5	30.5	31.5	32.5	33.4	34.4
84	25.5	26.5	27.5	28.5	29.4	30.4	31.3	32.3	33.2	34.2
86	25.4	26.4	27.3	28.3	29.2	30.2	31.2	32.2	33.1	34.1
88	25.2	26.2	27.2	28.2	29.1	30.1	31.0	32.0	33.0	34.0
90	25.1	26.1	27.0	28.0	29.0	30.0	30.9	31.9	32.9	33.9
92	25.0	26.0	26.9	27.9	28.9	29.9	30.8	31.8	32.7	33.7
94	24.9	25.9	26.8	27.8	28.8	29.8	30.7	31.7	32.6	33.6
96	24.7	25.7	26.7	27.7	28.6	29.6	30.5	31.5	32.5	33.5
98	24.6	25.6	26.6	27.6	28.5	29.5	30.4	31.4	32.3	33.3
100	24.5	25.5	26.4	27.4	28.3	29.3	30.3	31.3	32.2	33.2
102	24.4	25.4	26.3	27.3	28.2	29.2	30.2	31.2	32.1	33.0
104	24.3	25.3	26.2	27.1	28.1	29.1	30.0	31.0	31.9	32.9
106	24.2	25.2	26.1	27.0	28.0	29.0	29.9	30.9	31.8	32.7
108	24.0	25.0	25.9	26.9	27.8	28.8	29.7	30.7	31.6	32.6
110	23.9	24.9	25.8	26.8	27.7	28.7	29.6	30.6	31.5	32.5
112	23.8	24.8	25.7	26.7	27.6	28.6	29.5	30.4	31.3	32.3
114	23.7	24.7	25.6	26.6	27.5	28.4	29.3	30.3	31.2	32.2
116	23.6	24.6	25.5	26.4	27.3	28.3	29.2	30.2	31.1	32.1
118	23.4	24.4	25.3	26.3	27.2	28.2	29.1	30.1	31.0	32.0
120	23.3	24.3	25.2	26.2	27.1	28.1	29.0	30.0	30.9	31.9

Observed temperature in °F.	Observed degrees Baumé									
	37.0	38.0	39.0	40.0	41.0	42.0	43.0	44.0	45.0	46.0
	Corresponding degrees Baumè at 60°F.									
30	39.3	40.3	41.4	42.4	43.5	44.5	45.6	46.6	47.7	48.7
32	39.2	40.2	41.3	42.3	43.4	44.3	45.4	46.4	47.5	48.5
34	39.0	40.0	41.1	42.1	43.2	44.2	45.3	46.3	47.3	48.3
36	38.9	39.9	41.0	42.0	43.1	44.0	45.1	46.1	47.2	48.2
38	38.7	39.7	40.8	41.8	42.9	43.9	45.0	46.0	47.0	48.0
40	38.5	39.5	40.6	41.6	42.7	43.7	44.8	45.8	46.8	47.8
42	38.4	39.4	40.5	41.5	42.5	43.5	44.6	45.6	46.6	47.6
44	38.2	39.2	40.3	41.3	42.4	43.4	44.4	45.4	46.4	47.4
46	38.1	39.1	40.1	41.1	42.2	43.2	44.2	45.2	46.2	47.2
48	37.9	38.9	39.9	40.9	42.0	43.0	44.1	45.1	46.1	47.1
50	37.8	38.8	39.8	40.8	41.8	42.8	43.9	44.9	45.9	46.9
52	37.6	38.6	39.6	40.7	41.7	42.7	43.7	44.7	45.7	46.7
54	37.4	38.4	39.5	40.5	41.5	42.5	43.5	44.5	45.5	46.5
56	37.3	38.3	39.3	40.3	41.3	42.3	43.3	44.3	45.3	46.3
58	37.1	38.1	39.1	40.1	41.1	42.1	43.1	44.1	45.2	46.2
60	37.0	38.0	39.0	40.0	41.0	42.0	43.0	44.0	45.0	46.0
62	36.9	37.9	38.9	39.9	40.9	41.9	42.9	43.9	44.9	45.9
64	36.7	37.7	38.7	39.7	40.7	41.7	42.7	43.7	44.7	45.7
66	36.6	37.6	38.6	39.5	40.5	41.5	42.5	43.5	44.5	45.5
68	36.4	37.4	38.4	39.4	40.4	41.4	42.4	43.3	44.3	45.3
70	36.2	37.2	38.2	39.2	40.2	41.2	42.2	43.1	44.1	45.1
72	36.1	37.1	38.1	39.1	40.0	41.0	42.0	43.0	44.0	45.0
74	35.9	36.9	37.9	38.9	39.8	40.8	41.8	42.8	43.8	44.8
76	35.8	36.8	37.8	38.7	39.7	40.7	41.7	42.7	43.6	44.6
78	35.6	36.6	37.6	38.6	39.5	40.5	41.5	42.5	43.4	44.4
80	35.5	36.5	37.5	38.5	39.4	40.4	41.3	42.3	43.2	44.2
82	35.3	36.3	37.3	38.3	39.2	40.2	41.2	42.2	43.1	44.1
84	35.2	36.2	37.2	38.2	39.1	40.1	41.0	42.0	42.9	43.9
86	35.1	36.1	37.0	38.0	38.9	39.9	40.9	41.9	42.8	43.8
88	34.9	35.9	36.9	37.9	38.8	39.8	40.7	41.7	42.6	43.6
90	34.8	35.8	36.7	37.7	38.6	39.6	40.5	41.5	42.5	43.5
92	34.6	35.6	36.6	37.6	38.5	39.5	40.4	41.4	42.3	43.3
94	34.5	35.5	36.4	37.4	38.3	39.3	40.2	41.2	42.2	43.2
96	34.4	35.4	36.3	37.3	38.2	39.2	40.1	41.1	42.0	43.0
98	34.2	35.2	36.1	37.1	38.0	39.0	39.9	40.9	41.8	42.8
100	34.1	35.1	36.0	37.0	37.9	38.9	39.8	40.7	41.6	42.6
102	33.9	34.9	35.8	36.8	37.7	38.7	39.6	40.6	41.5	42.5
104	33.8	34.8	35.7	36.7	37.6	38.6	39.5	40.4	41.3	42.3
106	33.6	34.6	35.5	36.5	37.4	38.4	39.3	40.3	41.2	42.2
108	33.5	34.5	35.4	36.4	37.3	38.3	39.2	40.1	41.0	42.0
110	33.4	34.4	35.3	36.3	37.2	38.1	39.0	40.0	40.9	41.8
112	33.2	34.2	35.1	36.1	37.0	38.0	38.9	39.8	40.7	41.6
114	33.1	34.1	35.0	36.0	36.9	37.8	38.7	39.7	40.6	41.5
116	33.0	34.0	34.9	35.9	36.8	37.7	38.6	39.5	40.4	41.4
118	32.9	33.9	34.8	35.7	36.6	37.5	38.4	39.4	40.3	41.2
120	32.8	33.7	34.6	35.6	36.5	37.4	38.3	39.2	40.1	41.0

Observed temperature in °F.	Observed degrees Baumé									
	47.0	48.0	49.0	50.0	51.0	52.0	53.0	54.0	55.0	56.0
	Corresponding degrees Baumé at 60°F.									
30	49.8	50.8	51.9	53.0	54.1	55.1	56.2	57.3	58.4	59.4
32	49.6	50.6	51.7	52.8	53.9	54.9	56.0	57.1	58.2	59.2
34	49.4	50.4	51.5	52.6	53.7	54.7	55.8	56.8	57.9	58.9
36	49.3	50.3	51.4	52.4	53.5	54.5	55.6	56.6	57.7	58.7
38	49.1	50.1	51.2	52.2	53.3	54.3	55.4	56.4	57.5	58.5
40	48.9	49.9	51.0	52.0	53.0	54.1	55.2	56.2	57.2	58.2
42	48.7	49.7	50.8	51.8	52.8	53.8	54.9	56.0	57.0	58.0
44	48.5	49.5	50.6	51.6	52.6	53.6	54.7	55.7	56.8	57.8
46	48.3	49.3	50.4	51.4	52.4	53.4	54.5	55.5	56.5	57.5
48	48.1	49.1	50.2	51.2	52.2	53.2	54.2	55.2	56.3	57.3
50	47.9	48.9	50.0	51.0	52.0	53.0	54.0	55.0	56.1	57.1
52	47.7	48.7	49.8	50.8	51.8	52.8	53.8	54.8	55.9	56.9
54	47.6	48.6	49.6	50.6	51.6	52.6	53.6	54.6	55.6	56.6
56	47.4	48.4	49.4	50.4	51.4	52.4	53.4	54.4	55.4	56.4
58	47.2	48.2	49.2	50.2	51.2	52.2	53.2	54.2	55.2	56.2
60	47.0	48.0	49.0	50.0	51.0	52.0	53.0	54.0	55.0	56.0
62	46.9	47.9	48.8	49.8	50.8	51.8	52.8	53.8	54.8	55.8
64	46.7	47.7	48.6	49.6	50.6	51.6	52.6	53.6	54.6	55.6
66	46.5	47.5	48.4	49.4	50.4	51.4	52.4	53.4	54.4	55.4
68	46.3	47.3	48.3	49.3	50 3	51.3	52.2	53.2	54.2	55.2
70	46.1	47.1	48.1	49.1	50.1	51.1	52.0	53.0	54.0	55.0
72	46.0	47.0	47.9	48.9	49.9	50.9	51.8	52.8	53.8	54.8
74	45.8	46.8	47.7	48.7	49.7	50.7	51.6	52.6	53.5	54.5
76	45.6	46.6	47.5	48.5	49.5	50.5	51.4	52.4	53.3	54.3
78	45.4	46.4	47.3	48.3	49.3	50.3	51.2	52.2	53.1	54.1
80	45.2	46.2	47.2	48.2	49.1	50.1	51.0	52.0	52.9	53.9
82	45.1	46.1	47.0	48.0	48.9	49.9	50.8	51.8	52.7	53.7
84	44.9	45.9	46.8	47.8	48.7	49.7	50.6	51.6	52.5	53.5
86	44.7	45.7	46.6	47.6	48.5	49.5	50.4	51.4	52.3	53.3
88	44.5	45.5	46.4	47.4	48.3	49.3	50.2	51.2	52.1	53.1
90	44.4	45.4	46.3	47.3	48.2	49.2	50.1	51.0	51.9	52.9
92	44.2	45.2	46.1	47.1	48.0	49.0	49.9	50.9	51.8	52.7
94	44.1	45.1	46.0	46.9	47.8	48.8	49.7	50.7	51.6	52.5
96	43.9	44.9	45.8	46.7	47.6	48.6	49.5	50.5	51.4	52.3
98	43.7	44.7	45.6	46.6	47.5	48.4	49.3	50.3	51.2	52.1
100	43.5	44.5	45.4	46.4	47.3	48.3	49.2	50.1	51.0	51.9
102	43.4	44.3	45.2	46.2	47.1	48.1	49.0	49.9	50.8	51.7
104	43.2	44.1	45.0	46.0	46.9	47.9	48.8	49.7	50.6	51.5
106	43.1	44.0	44.9	45.8	46.7	47.7	48.6	49.5	50.4	51.3
108	42.9	43.9	44.8	45.7	46.6	47.5	48.4	49.4	50.3	51.2
110	42.7	43.7	44.6	45.6	46.5	47.4	48.3	49.2	50.1	51.0
112	42.5	43.5	44.4	45.4	46.3	47.2	48.1	49.0	49.9	50.8
114	42.4	43.4	44.3	45.3	46.2	47.1	48.0	48.8	49.7	50.6
116	42.3	43.3	44.2	45.1	46.0	46.9	47.8	48.6	49.5	50.4
118	42.1	43.1	44.0	44.9	45.8	46.7	47.6	48.4	49.3	50.2
120	41.9	42.9	43.8	44.7	45.6	46.5	47.4	48.2	49.1	50.0

Observed temperature in °F.	Observed degrees Baumé									
	57.0	58.0	59.0	60.0	61.0	62.0	63.0	64.0	65.0	66.0
	Corresponding degrees Baumé at 60°F.									
30	60.5	61.6	62.7	63.7	64.8	65.8	66.9	67.9	69.0	70.0
32	60.3	61.3	62.4	63.4	64.5	65.5	66.6	67.7	68.8	69.8
34	60.0	61.0	62.1	63.1	64.2	65.2	66.3	67.4	68.5	69.5
36	59.8	60.8	61.9	62.9	64.0	65.0	66.1	67.1	68.2	69.2
38	59.5	60.5	61.6	62.6	63.7	64.7	65.8	66.8	67.9	68.9
40	59.3	60.3	61.4	62.4	63.5	64.5	65.5	66.5	67.6	68.6
42	59.1	60.1	61.2	62.2	63.3	64.3	65.3	66.3	67.4	68.4
44	58.9	59.9	61.0	62.0	63.0	64.0	65.0	66.0	67.1	68.1
46	58.6	59.6	60.7	61.7	62.7	63.7	64.8	65.8	66.8	67.8
48	58.4	59.4	60.4	61.4	62.5	63.5	64.5	65.5	66.5	67.5
50	58.1	59.1	60.2	61.2	62.2	63.2	64.2	65.2	66.2	67.2
52	57.9	58.9	60.0	61.0	62.0	63.0	64.0	65.0	66.0	67.0
54	57.7	58.7	59.8	60.8	61.8	62.8	63.8	64.8	65.8	66.8
56	57.5	58.5	59.5	60.5	61.5	62.5	63.6	64.6	65.6	66.6
58	57.3	58.3	59.3	60.3	61.3	62.3	63.3	64.3	65.3	66.3
60	57.0	58.0	59.0	60.0	61.0	62.0	63.0	64.0	65.0	66.0
62	56.8	57.8	58.8	59.8	60.8	61.8	62.7	63.7	64.7	65.7
64	56.6	57.6	59.6	59.6	60.5	61.5	62.5	63.5	64.5	65.5
66	56.4	57.4	58.3	59.3	60.3	61.3	62.3	63.3	64.2	65.2
68	56.1	57.1	58.1	59.1	60.1	61.1	62.1	63.1	64.0	65.0
70	55.9	56.9	57.9	58.9	59.8	60.8	61.8	62.8	63.8	64.8
72	55.7	56.7	57.7	58.7	59.6	60.6	61.6	62.6	63.5	64.5
74	55.5	56.5	57.4	58.4	59.3	60.3	61.3	62.3	63.2	64.2
76	55.3	56.3	57.2	58.2	59.1	60.1	61.0	62.0	63.0	64.0
78	55.0	56.0	57.0	58.0	58.9	59.9	60.8	61.8	62.8	63.8
80	54.8	55.8	56.8	57.8	58.7	59.7	60.6	61.6	62.6	63.6
82	54.6	55.6	56.5	57.5	58.4	59.4	60.4	61.4	62.3	63.3
84	54.4	55.4	56.3	57.3	58.2	59.2	60.1	61.1	62.0	63.0
86	54.2	55.2	56.1	57.1	58.0	59.0	59.9	60.9	61.8	62.8
88	54.0	55.0	55.9	56.9	57.8	58.8	59.7	60.6	61.5	62.5
90	53.8	54.8	55.7	56.7	57.6	58.6	59.5	60.4	61.3	62.3
92	53.6	54.6	55.5	56.5	57.4	58.4	59.3	60.2	61.1	62.1
94	53.4	54.3	55.2	56.2	57.1	58.1	59.0	59.9	60.8	61.8
96	53.2	54.1	55.0	56.0	56.9	57.9	58.8	59.7	60.6	61.6
98	53.0	53.9	54.8	55.8	56.7	57.6	58.5	59.5	60.4	61.3
100	52.8	53.7	54.6	55.6	56.5	57.4	58.3	59.3	60.2	61.1
102	52.6	53.5	54.4	55.4	56.3	57.2	58.1	59.0	59.9	60.9
104	52.4	53.3	54.2	55.2	56.1	57.0	57.9	58.8	59.7	60.7
106	52.2	53.1	54.0	55.0	55.9	56.8	57.7	58.6	59.5	60.4
108	52.1	53.0	53.9	54.8	55.7	56.6	57.5	58.4	59.3	60.2
110	51.9	52.8	53.7	54.6	55.5	56.4	57.3	58.2	59.1	60.0
112	51.7	52.6	53.5	54.4	55.2	56.2	57.1	58.0	58.9	59.8
114	51.5	52.4	53.3	54.2	55.1	56.0	56.9	57.8	58.7	59.6
116	51.3	52.2	53.1	54.0	54.9	55.8	56.7	57.6	58.4	59.3
118	51.1	52.0	52.9	53.8	54.7	55.6	56.5	57.4	58.2	59.1
120	50.9	51.8	52.7	53.6	54.5	55.4	56.3	57.2	58.0	58.9

Observed temperature in °F.	Observed degrees Baumé									
	67.0	68.0	69.0	70.0	71.0	72.0	73.0	74.0	75.0	76.0
	Corresponding degrees Baumé at 60°F.									
30	71.1	72.1	73.2	74.3	75.4	76.4	77.5	78.5	79.6	80.7
32	70.9	71.9	73.0	74.0	75.1	76.1	77.2	78.2	79.3	80.4
34	70.6	71.6	72.7	73.7	74.8	75.8	76.9	77.9	79.0	80.1
36	70.3	71.3	72.4	73.4	74.5	75.5	76.6	77.6	78.7	79.7
38	70.0	71.0	72.1	73.1	74.2	75.2	76.3	77.3	78.4	79.4
40	69.7	70.7	71.8	72.8	73.9	74.9	76.0	77.0	78.1	79.1
42	69.4	70.4	71.5	72.5	73.6	74.6	75.7	76.7	77.8	78.8
44	69.1	70.1	71.2	72.2	73.3	74.3	75.4	76.4	77.5	78.5
46	68.8	69.8	70.9	71.9	73.0	74.0	75.1	76.1	77.1	78.1
48	68.6	69.6	70.6	71.6	72.7	73.7	74.8	75.8	76.8	77.8
50	68.3	69.3	70.4	71.4	72.5	73.5	74.5	75.5	76.5	77.5
52	68.0	69.0	70.1	71.1	72.2	73.2	74.2	75.2	76.2	77.2
54	67.8	68.8	69.9	70.9	71.9	72.9	73.9	74.9	75.9	76.9
56	67.6	68.6	69.6	70.6	71.6	72.6	73.6	74.6	75.6	76.6
58	67.3	68.3	69.3	70.3	71.3	72.3	73.3	74.3	75.3	76.3
60	67.0	68.0	69.0	70.0	71.0	72.0	73.0	74.0	75.0	76.0
62	66.7	67.7	68.7	69.7	70.7	71.7	72.7	73.7	74.7	75.7
64	66.4	67.4	68.4	69.4	70.4	71.4	72.4	73.4	74.4	75.4
66	66.2	67.2	68.2	69.2	70.1	71.1	72.1	73.1	74.1	75.1
68	66.0	67.0	67.9	68.9	69.8	70.8	71.8	72.8	73.8	74.8
70	65.7	66.7	67.6	68.6	69.5	70.5	71.5	72.5	73.5	74.5
72	65.4	66.4	67.4	68.4	69.3	70.3	71.2	72.2	73.2	74.2
74	65.2	66.2	67.2	68.2	69.1	70.1	71.0	72.0	72.9	73.9
76	64.9	65.9	66.9	67.9	68.8	69.8	70.8	71.8	72.7	73.7
78	64.7	65.6	66.6	67.6	68.5	69.5	70.5	71.5	72.4	73.4
80	64.5	65.4	66.4	67.4	68.3	69.3	70.2	71.2	72.1	73.1
82	64.2	65.2	66.1	67.1	68.0	69.0	69.9	70.9	71.8	72.8
84	63.9	64.9	65.8	66.8	67.7	68.7	69.6	70.6	71.5	72.5
86	63.7	64.7	65.6	66.6	67.5	68.4	69.3	70.3	71 3	72.3
88	63.4	64.4	65.3	66.3	67.2	68.2	69.1	70.1	71.0	72.0
90	63.2	64.2	65.1	66.1	67.0	68.0	68.9	69.9	70.8	71.7
92	63.0	64.0	64.9	65.8	66.7	67.7	68.6	69.6	70.5	71.4
94	62.7	63.7	64.6	65.6	66.5	67.4	68.3	69.3	70.2	71.1
96	62.5	63.5	64.4	65.4	66.3	67.2	68.1	69.0	69.9	70.8
98	62.2	63.2	64.1	65.1	66.0	66.9	67.8	68.8	69.7	70.6
100	62.0	63.0	63.9	64.9	65.8	66.7	67.6	68.5	69.4	70.4
102	61.8	62.8	63.7	64.6	65.5	66.4	67.3	68.2	69.1	70.1
104	61.6	62.5	63.4	64.3	65.2	66.1	67.0	67.9	68.8	69.8
106	61.3	62.3	63.2	64.1	65.0	65.9	66.8	67.7	68.6	69.5
108	61.1	62.0	62.9	63.8	64.8	65.7	66.6	67.5	68.4	69.3
110	60.9	61.8	62.7	63.6	64.5	65.4	66.3	67.2	68.1	69.0
112	60.7	61.6	62.5	63.3	64.2	65.2	66.1	67.0	67.8	68.7
114	60.5	61.4	62.3	63.1	64.0	64.9	65.8	66.7	67.6	68.5
116	60.2	61.1	62.0	62.9	63.8	64.7	65.6	66.5	67.4	68.3
118	60.0	60.9	61.8	62.7	63.6	64.5	65.4	66.3	67.1	68.0
120	59.8	60.7	61.6	62.5	63.3	64.2	65.1	66.0	66.8	67.7

Observed temperature in °F.	Observed degrees Baumé									
	77.0	78.0	79.0	80.0	81.0	82.0	83.0	84.0	85.0	86.0
	Corresponding degrees Baumé at 60°F.									
30	81.8	82.9	84.0	85.0	86.1	87.1	88.2	89.3	90.4	91.5
32	81.5	82.6	83.7	84.7	85.8	86.8	87.9	89.0	90.1	91.1
34	81.2	82.2	83.3	84.3	85.4	86.4	87.5	88.6	89.7	90.7
36	80.8	81.9	83.0	84.0	85.1	86.1	87.2	88.2	89.3	90.3
38	80.5	81.5	82.6	83.6	84.7	85.7	86.8	87.8	88.9	89.9
40	80.1	81.1	82.2	83.2	84.3	85.3	86.4	87.4	88.5	89.5
42	79.8	80.8	81.9	82.9	84.0	85.0	86.1	87.1	88.2	89.2
44	79.5	80.5	81.6	82.6	83.7	84.7	85.8	86.8	87.8	88.8
46	79.2	80.2	81.3	82.3	83.4	84.4	85.4	86.5	87.5	88.5
48	78.9	79.9	81.0	82.0	83.0	84.0	85.1	86.1	87.1	88.1
50	78.6	79.6	80.6	81.6	82.6	83.6	84.7	85.7	86.7	87.7
52	78.2	79.2	80.3	81.3	82.3	83.3	84.3	85.3	86.3	87.3
54	77.9	78.9	79.9	81.0	82.0	83.0	84.0	85.0	86.0	87.0
56	77.6	78.6	79.6	80.6	81.6	82.6	83.7	84.7	85.7	86.7
58	77.3	78.3	79.3	80.3	81.3	82.3	83.3	84.3	85.3	86.3
60	77.0	78.0	79.0	80.0	81.0	82.0	83.0	84.0	85.0	86.0
62	76.7	77.7	78.7	79.7	80.7	81.7	82.7	83.7	84.7	85.7
64	76.4	77.4	78.4	79.4	80.4	81.4	82.3	83.4	84.3	85.3
66	76.1	77.1	78.1	79.1	80.0	81.0	82.0	83.0	84.0	85.0
68	75.8	76.8	77.7	78.7	79.7	80.7	81.7	82.7	83.7	84.7
70	75.5	76.5	77.4	78.4	79.4	80.4	81.4	82.4	83.3	84.3
72	75.2	76.2	77.1	78.1	79.1	80.1	81.1	82.1	83.0	84.0
74	74.9	75.9	76.8	77.8	78.8	79.8	80.7	81.7	82.7	83.7
76	74.6	75.6	76.5	77.5	78.4	79.4	80.4	81.4	82.4	83.4
78	74.3	75.3	76.2	77.2	78.1	79.1	80.1	81.1	82.0	83.0
80	74.0	75.0	75.9	76.9	77.8	78.8	79.8	80.8	81.7	82.7
82	73.7	74.7	75.6	76.6	77.5	78.5	79.4	80.4	81.3	82.3
84	73.4	74.5	75.3	76.3	77.2	78.2	79.1	80.1	81.0	82.0
86	73.2	74.1	75.0	76.0	76.9	77.9	78.8	79.8	80.7	81.7
88	72.9	73.9	74.8	75.8	76.7	77.6	78.5	79.5	80.4	81.4
90	72.6	73.6	74.5	75.5	76.4	77.3	78.2	79.2	80.1	81.1
92	72.3	73.3	74.2	75.2	76.1	77.0	77.9	78.9	79.8	80.8
94	72.0	73.0	73.9	74.9	75.8	76.7	77.6	78.6	79.5	80.5
96	71.7	72.7	73.6	74.6	75.5	76.4	77.3	78.3	79.2	80.2
98	71.5	72.4	73.3	74.3	75.2	76.1	77.0	78.0	78.9	79.8
100	71.2	72.1	73.0	74.0	74.9	75.8	76.7	77.6	78.5	79.5
102	71.0	71.9	72.8	73.7	74.6	75.5	76.4	77.3	78.2	79.2
104	70.7	71.6	72.5	73.4	74.3	75.2	76.1	77.0	77.9	78.8
106	70.4	71.3	72.2	73.1	74.0	74.9	75.8	76.7	77.6	78.5
108	70.1	71.0	71.9	72.8	73.7	74.6	75.5	76.4	77.3	78.2
110	69.8	70.7	71.6	72.5	73.4	74.3	75.2	76.1	77.0	77.9
112	69.6	70.5	71.4	72.3	73.2	74.1	74.9	75.8	76.7	77.6
114	69.4	70.3	71.2	72.1	72.9	73.8	74.6	75.5	76.4	77.3
116	69.1	70.0	70.9	71.8	72.6	73.5	74.3	75.2	76.1	77.0
118	68.8	69.7	70.6	71.5	72.3	73.2	74.0	74.9	75.8	76.7
120	68.5	69.4	70.3	71.2	72.0	72.9	73.7	74.6	75.5	76.4

Observed temperature in °F.	Observed degrees Baumé									
	87.0	88.0	89.0	90.0	91.0	92.0	93.0	94.0	95.0	96.0
	Corresponding degrees Baumé at 60°F.									
30	92.6	93.6	94.7	95.7						
32	92.2	93.2	94.3	95.3						
34	91.8	92.9	93.9	94.9	95.9					
36	91.4	92.5	93.6	94.6	95.6					
38	91.0	92.1	93.2	94.2	95.2					
40	90.6	91.7	92.8	93.8	94.9	95.9				
42	90.3	91.3	92.4	93.4	94.5	95.5				
44	89.9	90.9	92.0	93.0	94.1	95.1	96.1			
46	89.6	90.6	91.7	92.7	93.7	94.7	95.7			
48	89.2	90.2	91.3	92.3	93.3	94.3	95.3			
50	88.8	89.8	90.9	91.9	92.9	93.9	94.9	95.9		
52	88.4	89.4	90.5	91.5	92.5	93.5	94.5	95.5		
54	88.0	89.0	90.1	91.1	92.1	93.1	94.1	95.1		
56	87.7	88.7	89.7	90.7	91.7	92.7	93.7	94.7	95.7	
58	87.3	88.3	89.4	90.4	91.4	92.4	93.4	94.4	95.4	
60	87.0	88.0	89.0	90.0	91.0	92.0	93.0	94.0	95.0	96.0
62	86.7	87.7	88.6	89.6	90.6	91.6	92.6	93.6	94.6	95.6
64	86.3	87.3	88.3	89.3	90.3	91.3	92.2	93.2	94.2	95.2
66	86.0	87.0	88.0	89.0	89.9	90.9	91.8	92.8	93.8	94.8
68	85.6	86.6	87.6	88.6	89.5	90.5	91.4	92.4	93.4	94.4
70	85.3	86.3	87.3	88.3	89.2	90.1	91.0	92.0	93.0	94.0
72	85.0	86.0	86.9	87.9	88.8	89.8	90.7	91.7	92.7	93.7
74	84.6	85.6	86.5	87.5	88.4	89.4	90.3	91.3	92.3	93.3
76	84.3	85.3	86.2	87.2	88.1	89.1	90.0	91.0	92.0	93.0
78	84.0	85.0	85.9	86.9	87.8	88.7	89.6	90.6	91.6	92.6
80	83.6	84.6	85.5	86.5	87.4	88.4	89.3	90.2	91.2	92.2
82	83.2	84.2	85.1	86.1	87.0	88.0	88.9	89.8	90.8	91.8
84	82.9	83.8	84.7	85.7	86.6	87.6	88.5	89.4	90.4	91.4
86	82.6	83.5	84.4	85.4	86.3	87.3	88.2	89.1	90.0	91.0
88	82.3	83.2	84.1	85.1	86.0	87.0	87.9	88.8	89.7	90.7
90	82.0	82.9	83.8	84.8	85.7	86.6	87.5	88.4	89.3	90.3
92	81.7	82.6	83.5	84.4	85.3	86.2	87.1	88.1	89.0	90.0
94	81.3	82.2	83.1	84.1	85.0	85.9	86.8	87.7	88.6	89.6
96	81.0	81.9	82.8	83.7	84.6	85.6	86.5	87.4	88.3	89.3
98	80.7	81.6	82.5	83.4	84.3	85.2	86.1	87.0	88.0	89.0
100	80.4	81.3	82.2	83.1	84.0	84.9	85.8	86.7	87.6	88.6
102	80.1	81.0	81.9	82.8	83.7	84.6	85.5	86.4	87.3	88.3
104	79.7	80.6	81.5	82.5	83.4	84.3	85.2	86.1	87.0	87.9
106	79.4	80.3	81.2	82.1	83.0	83.9	84.8	85.7	86.6	87.6
108	79.1	80.0	80.9	81.8	82.7	83.6	84.5	85.4	86.3	87.2
110	78.8	79.7	80.6	81.5	82.4	83.3	84.2	85.1	86.0	86.9
112	78.5	79.4	80.3	81.2	82.1	83.0	83.8	84.7	85.6	86.6
114	78.2	79.1	80.0	80.9	81.7	82.6	83.5	84.4	85.3	86.2
116	77.9	78.8	79.7	80.6	81.4	82.3	83.2	84.1	85.0	85.9
118	77.5	78.4	79.3	80.2	81.1	82.0	82.8	83.7	84.6	85.6
120	77.2	78.1	79.0	79.9	80.8	81.7	82.5	83.4	84.3	85.2

Degrees Baumé, Pounds Per Gallon, and Gallons per Pound, Corresponding to the Various Specific Gravities Designated. (From Bureau of Standards Circular 57.)

Specific gravity at 60°/60°F.	Degrees Baumé	Pounds per gallon	Gallons per pound	Specific gravity at 60°/60°F.	Degrees Baumé	Pounds per gallon	Gallons per pound
0.600	103.33	4.993	0.2003	0.650	85.38	5.410	0.1848
0.601	102.94	5.001	0.1999	0.651	85.05	5.418	0.1846
0.602	102.56	5.010	0.1996	0.652	84.72	5.426	0.1843
0.603	102.17	5.018	0.1993	0.653	84.40	5.435	0.1840
0.604	101.79	5.026	0.1990	0.654	84.07	5.443	0.1837
0.605	101.40	5.035	0.1986	0.655	83.74	5.452	0.1834
0.606	101.02	5.043	0.1983	0.656	83.42	5.460	0.1832
0.607	100.64	5.051	0.1980	0.657	83.09	5.468	0.1829
0.608	100.26	5.060	0.1976	0.658	82.77	5.476	0.1826
0.609	99.88	5.068	0.1973	0.659	82.44	5.485	0.1823
0.610	99.51	5.076	0.1970	0.660	82.12	5.493	0.1820
0.611	99.13	5.084	0.1967	0.661	81.80	5.502	0.1818
0.612	98.76	5.093	0.1963	0.662	81.48	5.510	0.1815
0.613	98.38	5.101	0.1960	0.663	81.16	5.518	0.1812
0.614	98.01	5.110	0.1957	0.664	80.84	5.526	0.1810
0.615	97.64	5.118	0.1954	0.665	80.53	5.535	0.1807
0.616	97.27	5.126	0.1951	0.666	80.21	5.543	0.1804
0.617	96.90	5.135	0.1948	0.667	79.90	5.552	0.1801
0.618	96.54	5.143	0.1944	0.668	79.58	5.560	0.1799
0.619	96.17	5.151	0.1941	0.669	79.27	5.568	0.1796
0.620	95.81	5.160	0.1938	0.670	78.96	5.577	0.1793
0.621	95.44	5.168	0.1935	0.671	78.64	5.585	0.1790
0.622	95.08	5.176	0.1932	0.672	78.33	5.593	0.1788
0.623	94.72	5.185	0.1929	0.673	78.02	5.602	0.1785
0.624	94.36	5.193	0.1926	0.674	77.72	5.610	0.1782
0.625	94.00	5.201	0.1923	0.675	77.41	5.618	0.1780
0.626	93.64	5.210	0.1920	0.676	77.10	5.627	0.1777
0.627	93.28	5.218	0.1916	0.677	76.80	5.635	0.1775
0.628	92.93	5.226	0.1913	0.678	76.49	5.643	0.1772
0.629	92.58	5.235	0.1910	0.679	76.19	5.652	0.1769
0.630	92.22	5.243	0.1907	0.680	75.88	5.660	0.1767
0.631	91.87	5.251	0.1904	0.681	75.58	5.668	0.1764
0.632	91.52	5.260	0.1901	0.682	75.28	5.677	0.1762
0.633	91.17	5.268	0.1898	0.683	74.98	5.685	0.1759
0.634	90.82	5.276	0.1895	0.684	74.68	5.693	0.1756
0.635	90.47	5.285	0.1892	0.685	74.38	5.702	0.1754
0.636	90.13	5.293	0.1889	0.686	74.08	5.710	0.1751
0.637	89.78	5.301	0.1886	0.687	73.78	5.718	0.1749
0.638	89.44	5.310	0.1883	0.688	73.49	5.727	0.1746
0.639	89.09	5.318	0.1880	0.689	73.19	5.735	0.1744
0.640	88.75	5.326	0.1877	0.690	72.90	5.743	0.1741
0.641	88.41	5.335	0.1874	0.691	72.60	5.752	0.1739
0.642	88.07	5.343	0.1872	0.692	72.31	5.760	0.1736
0.643	87.73	5.351	0.1869	0.693	72.02	5.768	0.1734
0.644	87.39	5.360	0.1866	0.694	71.73	5.777	0.1731
0.645	87.05	5.368	0.1863	0.695	71.44	5.785	0.1729
0.646	86.72	5.376	0.1860	0.696	71.15	5.793	0.1726
0.647	86.38	5.385	0.1857	0.697	70.86	5.802	0.1724
0.648	86.05	5.393	0.1854	0.698	70.57	5.810	0.1721
0.649	85.72	5.402	0.1851	0.699	70.29	5.818	0.1719

DEGREES BAUMÉ, POUNDS PER GALLON, AND GALLONS PER POUND, CORRESPONDING TO THE VARIOUS SPECIFIC GRAVITIES DESIGNATED (*Continued*)

Specific gravity at 60°/60°F.	Degrees Baumé	Pounds per gallon	Gallons per pound	Specific gravity at 60°/60°F.	Degrees Baumé	Pounds per gallon	Gallons per pound
0.700	70.00	5.827	0.1716	0.750	56.67	6.244	0.1602
0.701	69.72	5.835	0.1714	0.751	56.42	6.252	0.1600
0.702	69.43	5.843	0.1711	0.752	56.17	6.260	0.1597
0.703	69.15	5.852	0.1709	0.753	55.92	6.269	0.1595
0.704	68.86	5.860	0.1706	0.754	55.68	6.277	0.1593
0.705	68.58	5.868	0.1704	0.755	55.43	6.285	0.1591
0.706	68.30	5.877	0.1702	0.756	55.18	6.294	0.1589
0.707	68.02	5.885	0.1699	0.757	54.94	6.302	0.1587
0.708	67.74	5.894	0.1697	0.758	54.70	6.310	0.1585
0.709	67.46	5.902	0.1694	0.759	54.45	6.319	0.1583
0.710	67.18	5.910	0.1692	0.760	54.21	6.327	0.1580
0.711	66.91	5.918	0.1790	0.761	53.97	6.335	0.1578
0.712	66.63	5.927	0.1687	0.762	53.73	6.344	0.1576
0.713	66.35	5.935	0.1685	0.763	53.49	6.352	0.1574
0.714	66.08	5.944	0.1682	0.764	53.25	6.360	0.1572
0.715	65.80	5.952	0.1680	0.765	53.01	6.369	0.1570
0.716	65.53	5.960	0.1678	0.766	52.77	6.377	0.1568
0.717	65.26	5.968	0.1676	0.767	52.53	6.386	0.1566
0.718	64.99	5.977	0.1673	0.768	52.29	6.394	0.1564
0.719	64.72	5.985	0.1671	0.769	52.06	6.402	0.1562
0.720	64.44	5.994	0.1668	0.770	51.82	6.410	0.1560
0.721	64.18	6.002	0.1666	0.771	51.58	6.419	0.1558
0.722	63.91	6.010	0.1664	0.772	51.35	6.417	0.1556
0.723	63.64	6.018	0.1662	0.773	51.11	6.436	0.1554
0.724	63.37	6.027	0.1659	0.774	50.88	6.444	0.1552
0.725	63.10	6.035	0.1657	0.775	50.64	6.452	0.1550
0.726	62.84	6.044	0.1655	0.776	50.41	6.460	0.1548
0.727	62.57	6.052	0.1652	0.777	50.18	6.469	0.1546
0.728	62.31	6.060	0.1650	0.778	49.95	6.477	0.1544
0.729	62.04	6.068	0.1648	0.779	49.72	6.486	0.1542
0.730	61.78	6.077	0.1646	0.780	49.49	6.494	0.1540
0.731	61.52	6.085	0.1643	0.781	49.26	6.502	0.1538
0.732	61.26	6.094	0.1641	0.782	49.03	6.510	0.1536
0.733	61.00	6.102	0.1639	0.783	48.80	6.519	0.1534
0.734	60.74	6.110	0.1637	0.784	48.57	6.527	0.1532
0.735	60.48	6.119	0.1634	0.785	48.34	6.536	0.1530
0.736	60.22	6.127	0.1632	0.786	48.12	6.544	0.1528
0.737	59.96	6.135	0.1630	0.787	47.89	6.552	0.1526
0.738	59.70	6.144	0.1628	0.788	47.66	6.560	0.1524
0.739	59.44	6.152	0.1626	0.789	47.44	6.569	0.1522
0.740	59.19	6.160	0.1623	0.790	47.22	6.577	0.1520
0.741	58.93	6.169	0.1621	0.791	46.99	6.586	0.1518
0.742	58.68	6.177	0.1619	0.792	46.77	6.594	0.1517
0.743	58.42	6.185	0.1617	0.793	46.54	6.602	0.1515
0.744	58.17	6.194	0.1615	0.794	46.32	6.611	0.1513
0.745	57.92	6.202	0.1612	0.795	46.10	6.619	0.1511
0.746	57.67	6.210	0.1610	0.796	45.88	6.627	0.1509
0.747	57.42	6.219	0.1608	0.797	45.66	6.636	0.1507
0.748	57.17	6.227	0.1606	0.798	45.44	6.644	0.1505
0.749	56.92	6.235	0.1604	0.799	45.22	6.652	0.1503

DEGREES BAUMÉ, POUNDS PER GALLON, AND GALLONS PER POUND, CORRESPONDING TO THE VARIOUS SPECIFIC GRAVITIES DESIGNATED (*Continued*)

Specific gravity at 60°/60°F.	Degrees Baumé	Pounds per gallon	Gallons per pound	Specific gravity at 60°/60°F.	Degrees Baumé	Pounds per gallon	Gallons per pound
0.800	45.00	6.661	0.1501	0.850	34.71	7.078	0.1413
0.801	44.78	6.669	0.1500	0.851	34.51	7.086	0.1411
0.802	44.56	6.677	0.1498	0.852	34.32	7.094	0.1410
0.803	44.35	6.686	0.1496	0.853	34.13	7.103	0.1408
0.804	44.13	6.694	0.1494	0.854	33.93	7.111	0.1406
0.805	43.91	6.702	0.1492	0.855	33.74	7.119	0.1405
0.806	43.70	6.711	0.1490	0.856	33.55	7.128	0.1403
0.807	43.48	6.719	0.1488	0.857	33.36	7.136	0.1401
0.808	43.27	6.727	0.1486	0.858	33.17	7.144	0.1400
0.809	43.05	6.736	0.1485	0.859	32.98	7.153	0.1398
0.810	42.84	6.744	0.1483	0.860	32.79	7.161	0.1396
0.811	42.63	6.752	0.1481	0.861	32.60	7.169	0.1395
0.812	42.41	6.761	0.1479	0.862	32.41	7.178	0.1393
0.813	42.20	6.769	0.1477	0.863	32.22	7.186	0.1392
0.814	41.99	6.777	0.1476	0.864	32.04	7.194	0.1390
0.815	41.78	6.786	0.1474	0.865	31.85	7.203	0.1388
0.816	41.57	6.794	0.1472	0.866	31.66	7.211	0.1387
0.817	41.36	6.802	0.1470	0.867	31.48	7.219	0.1385
0.818	41.15	6.811	0.1468	0.868	31.29	7.228	0.1384
0.819	40.94	6.819	0.1466	0.869	31.10	7.236	0.1382
0.820	40.73	6.827	0.1465	0.870	30.92	7.244	0.1380
0.821	40.52	6.836	0.1463	0.871	30.74	7.253	0.1379
0.822	40.32	6.844	0.1461	0.872	30.55	7.261	0.1377
0.823	40.11	6.852	0.1459	0.873	30.37	7.269	0.1376
0.824	39.90	6.861	0.1458	0.874	30.18	7.278	0.1374
0.825	39.70	6.869	0.1456	0.875	30.00	7.286	0.1372
0.826	39.49	6.877	0.1454	0.876	29.82	7.294	0.1371
0.827	39.29	6.886	0.1452	0.877	29.64	7.303	0.1369
0.828	39.08	6.894	0.1450	0.878	29.45	7.311	0.1368
0.829	38.88	6.902	0.1449	0.879	29.27	7.319	0.1366
0.830	38.68	6.911	0.1447	0.880	29.09	7.328	0.1365
0.831	38.47	6.919	0.1445	0.881	28.91	7.336	0.1363
0.832	38.27	6.927	0.1444	0.882	28.73	7.344	0.1362
0.833	38.07	6.936	0.1442	0.883	28.55	7.353	0.1360
0.834	37.87	6.944	0.1440	0.884	28.37	7.361	0.1358
0.835	37.66	6.952	0.1438	0.885	28.19	7.369	0.1357
0.836	37.46	6.961	0.1437	0.886	28.01	7.378	0.1355
0.837	37.26	6.969	0.1435	0.887	27.84	7.386	0.1354
0.838	37.06	6.978	0.1433	0.888	27.66	7.394	0.1352
0.839	36.87	6.986	0.1432	0.889	27.48	7.403	0.1351
0.840	36.67	6.994	0.1430	0.890	27.30	7.411	0.1349
0.841	36.47	7.002	0.1428	0.891	27.13	7.419	0.1348
0.842	36.27	7.011	0.1426	0.892	26.95	7.428	0.1346
0.843	36.07	7.019	0.1425	0.893	26.78	7.436	0.1345
0.844	35.88	7.028	0.1423	0.894	26.60	7.444	0.1343
0.845	35.68	7.036	0.1421	0.895	26.42	7.453	0.1342
0.846	35.48	7.044	0.1420	0.896	26.25	7.461	0.1340
0.847	35.29	7.052	0.1418	0.897	26.08	7.469	0.1339
0.848	35.09	7.061	0.1416	0.898	25.90	7.478	0.1337
0.849	34.90	7.069	0.1415	0.899	25.73	7.486	0.1336

DEGREES BAUMÉ, POUNDS PER GALLON, AND GALLONS PER POUND, CORRESPONDING TO THE VARIOUS SPECIFIC GRAVITIES DESIGNATED (*Continued*)

Specific gravity at 60°/60°F.	Degrees Baumé	Pounds per gallon	Gallons per pound	Specific gravity at 60°/60°F.	Degrees Baumé	Pounds per gallon	Gallons per pound
0.900	25.56	7.494	0.1334	0.950	17.37	7.911	0.1264
0.901	25.38	7.503	0.1333	0.951	17.21	7.920	0.1263
0.902	25.21	7.511	0.1331	0.952	17.06	7.928	0.1261
0.903	25.04	7.519	0.1330	0.953	16.90	7.937	0.1260
0.904	24.87	7.528	0.1328	0.954	16.75	7.945	0.1259
0.905	24.70	7.536	0.1327	0.955	16.60	7.953	0.1257
0.906	24.52	7.544	0.1326	0.956	16.44	7.962	0.1256
0.907	24.36	7.553	0.1324	0.957	16.29	7.970	0.1255
0.908	24.18	7.561	0.1323	0.958	16.14	7.978	0.1253
0.909	24.02	7.569	0.1321	0.959	15.98	7.987	0.1252
0.910	23.85	7.578	0.1320	0.960	15.83	7.995	0.1251
0.911	23.68	7.586	0.1318	0.961	15.68	8.003	0.1250
0.912	23.51	7.594	0.1317	0.962	15.53	8.012	0.1248
0.913	23.34	7.603	0.1315	0.963	15.38	8.020	0.1247
0.914	23.17	7.611	0.1314	0.964	15.23	8.028	0.1246
0.915	23.00	7.620	0.1312	0.965	15.08	8.036	0.1244
0.916	22.84	7.628	0.1311	0.966	14.93	8.045	0.1243
0.917	22.67	7.636	0.1310	0.967	14.78	8.053	0.1242
0.918	22.51	7.645	0.1308	0.968	14.63	8.062	0.1240
0.919	22.34	7.653	0.1307	0.969	14.48	8.070	0.1239
0.920	22.17	7.661	0.1305	0.970	14.33	8.078	0.1238
0.921	22.01	7.670	0.1304	0.971	14.18	8.087	0.1237
0.922	21.84	7.678	0.1302	0.972	14.03	8.095	0.1235
0.923	21.68	7.686	0.1301	0.973	13.88	8.103	0.1234
0.924	21.52	7.695	0.1300	0.974	13.74	8.112	0.1233
0.925	21.35	7.703	0.1298	0.975	13.59	8.120	0.1232
0.926	21.19	7.711	0.1297	0.976	13.44	8.128	0.1230
0.927	21.02	7.720	0.1295	0.977	13.30	8.137	0.1229
0.928	20.86	7.728	0.1294	0.978	13.15	8.145	0.1228
0.929	20.70	7.736	0.1293	0.979	13.00	8.153	0.1227
0.930	20.54	7.745	0.1291	0.980	12.86	8.162	0.1225
0.931	20.38	7.753	0.1290	0.981	12.71	8.170	0.1224
0.932	20.22	7.761	0.1288	0.982	12.57	8.178	0.1223
0.933	20.05	7.770	0.1287	0.983	12.42	8.187	0.1221
0.934	19.89	7.778	0.1286	0.984	12.28	8.195	0.1220
0.935	19.73	7.786	0.1284	0.985	12.13	8.203	0.1219
0.936	19.57	7.795	0.1283	0.986	11.99	8.212	0.1218
0.937	19.41	7.803	0.1282	0.987	11.84	8.220	0.1217
0.938	19.25	7.811	0.1280	0.988	11.70	8.228	0.1215
0.939	19.10	7.820	0.1279	0.989	11.56	8.237	0.1214
0.940	18.94	7.828	0.1278	0.990	11.41	8.245	0.1213
0.941	18.78	7.836	0.1276	0.991	11.27	8.253	0.1212
0.942	18.62	7.845	0.1275	0.992	11.13	8.262	0.1210
0.943	18.46	7.853	0.1273	0.993	10.99	8.270	0.1209
0.944	18.30	7.861	0.1272	0.994	10.84	8.278	0.1208
				0.995	10.70	8.287	0.1207
0.945	18.15	7.870	0.1271	0.996	10.56	8.295	0.1206
0.946	17.99	7.878	0.1269	0.997	10.42	8.303	0.1204
0.947	17.84	7.886	0.1268	0.998	10.28	8.312	0.1203
0.948	17.68	7.895	0.1267	0.999	10.14	8.320	0.1202
0.949	17.52	7.903	0.1265	1.000	10.00	8.328	0.1201

Specific Gravities, Pounds per Gallon, and Gallons per Pound, Corresponding to the Various Degrees Baumé Designated. (From Bureau of Standards Circular 57.)

Degrees Baumé	Specific gravity at 60°/60°F.	Pounds per gallon	Gallons per pound	Degrees Baumé	Specific gravity at 60°/60°F.	Pounds per gallon	Gallons per pound
10.0	1.0000	8.328	0.1201	16.0	0.9589	7.986	0.1252
10.1	0.9993	8.322	0.1202	16.1	0.9582	7.980	0.1253
10.2	0.9986	8.317	0.1202	16.2	0.9576	7.975	0.1254
10.3	0.9979	8.311	0.1203	16.3	0.9569	7.969	0.1255
10.4	0.9972	8.305	0.1204	16.4	0.9563	7.964	0.1256
10.5	0.9964	8.299	0.1205	16.5	0.9556	7.959	0.1256
10.6	0.9957	8.293	0.1206	16.6	0.9550	7.953	0.1257
10.7	0.9950	8.287	0.1207	16.7	0.9543	7.948	0.1258
10.8	0.9943	8.281	0.1208	16.8	0.9537	7.942	0.1259
10.9	0.9936	8.275	0.1208	16.9	0.9530	7.937	0.1260
11.0	0.9929	8.269	0.1209	17.0	0.9524	7.931	0.1261
11.1	0.9922	8.263	0.1210	17.1	0.9517	7.926	0.1262
11.2	0.9915	8.258	0.1211	17.2	0.9511	7.921	0.1262
11.3	0.9908	8.252	0.1212	17.3	0.9504	7.915	0.1263
11.4	0.9901	8.246	0.1213	17.4	0.9498	7.910	0.1264
11.5	0.9894	8.240	0.1214	17.5	0.9492	7.904	0.1265
11.6	0.9887	8.234	0.1214	17.6	0.9485	7.899	0.1266
11.7	0.9880	8.228	0.1215	17.7	0.9479	7.894	0.1267
11.8	0.9873	8.223	0.1216	17.8	0.9472	7.888	0.1268
11.9	0.9866	8.217	0.1217	17.9	0.9466	7.883	0.1269
12.0	0.9859	8.211	0.1218	18.0	0.9459	8.877	0.1270
12.1	0.9852	8.205	0.1219	18.1	0.9453	7.872	0.1270
12.2	0.9845	8.199	0.1220	18.2	0.9447	7.867	0.1271
12.3	0.9838	8.194	0.1220	18.3	0.9440	7.861	0.1272
12.4	0.9831	8.188	0.1221	18.4	0.9434	7.856	0.1273
12.5	0.9825	8.182	0.1222	18.5	0.9428	7.851	0.1274
12.6	0.9818	8.176	0.1223	18.6	0.9421	7.846	0.1275
12.7	0.9811	8.171	0.1224	18.7	0.9415	7.841	0.1275
12.8	0.9804	8.165	0.1225	18.8	0.9409	7.835	0.1276
12.9	0.9797	8.159	0.1226	18.9	0.9402	7.830	0.1277
13.0	0.9790	8.153	0.1227	19.0	0.9396	7.825	0.1278
13.1	0.9783	8.148	0.1227	19.1	0.9390	7.820	0.1279
13.2	0.9777	8.142	0.1228	19.2	0.9383	7.814	0.1280
13.3	0.9770	8.137	0.1229	19.3	0.9377	7.809	0.1281
13.4	0.9763	8.131	0.1230	19.4	0.9371	7.804	0.1281
13.5	0.9756	8.125	0.1231	19.5	0.9365	7.799	0.1282
13.6	0.9749	8.119	0.1232	19.6	0.9358	7.793	0.1283
13.7	0.9743	8.114	0.1232	19.7	0.9352	7.788	0.1284
13.8	0.9736	8.108	0.1233	19.8	0.9346	7.783	0.1285
13.9	0.9729	8.102	0.1234	19.9	0.9340	7.778	0.1286
14.0	0.9722	8.096	0.1235	20.0	0.9333	7.772	0.1287
14.1	0.9715	8.091	0.1236	20.1	0.9327	7.767	0.1287
14.2	0.9709	8.086	0.1237	20.2	0.9321	7.762	0.1288
14.3	0.9702	8.080	0.1238	20.3	0.9315	7.757	0.1289
14.4	0.9695	8.074	0.1239	20.4	0.9309	7.752	0.1290
14.5	0.9688	8.069	0.1239	20.5	0.9302	7.747	0.1291
14.6	0.9682	8.063	0.1240	20.6	0.9296	7.742	0.1292
14.7	0.9675	8.058	0.1241	20.7	0.9290	7.736	0.1293
14.8	0.9669	8.052	0.1242	20.8	0.9284	7.731	0.1293
14.9	0.9662	8.047	0.1243	20.9	0.9278	7.726	0.1294
15.0	0.9655	8.041	0.1244	21.0	0.9272	7.721	0.1295
15.1	0.9649	8.035	0.1245	21.1	0.9265	7.716	0.1296
15.2	0.9642	8.030	0.1245	21.2	0.9259	7.711	0.1297
15.3	0.9635	8.024	0.1246	21.3	0.9253	7.706	0.1298
15.4	0.9629	8.019	0.1247	21.4	0.9247	7.701	0.1299
15.5	0.9622	8.013	0.1248	21.5	0.9241	7.696	0.1299
15.6	0.9615	8.007	0.1249	21.6	0.9235	7.690	0.1300
15.7	0.9609	8.002	0.1250	21.7	0.9229	7.685	0.1301
15.8	0.9602	7.997	0.1250	21.8	0.9223	7.680	0.1302
15.9	0.9596	7.991	0.1251	21.9	0.9217	7.675	0.1303

Degrees Baumé	Specific gravity at 60°/60°F.	Pounds per gallon	Gallons per pound	Degrees Baumé	Specific gravity at 60°/60°F.	Pounds per gallon	Gallons per pound
22.0	0.9211	7.670	0.1304	29.0	0.8805	7.332	0.1364
22.1	0.9204	7.665	0.1305	29.1	0.8799	7.328	0.1365
22.2	0.9198	7.660	0.1305	29.2	0.8794	7.323	0.1366
22.3	0.9192	7.655	0.1306	29.3	0.8788	7.318	0.1366
22.4	0.9186	7.650	0.1307	29.4	0.8783	7.314	0.1367
22.5	0.9180	7.645	0.1308	29.5	0.8777	7.309	0.1368
22.6	0.9174	7.640	0.1309	29.6	0.8772	7.305	0.1369
22.7	0.9168	7.635	0.1310	29.7	0.8766	7.300	0.1370
22.8	0.9162	7.630	0.1311	29.8	0.8761	7.295	0.1371
22.9	0.9156	7.625	0.1312	29.9	0.8755	7.291	0.1372
23.0	0.9150	7.620	0.1313	30.0	0.8750	7.286	0.1373
23.1	0.9144	7.615	0.1313	30.1	0.8745	7.282	0.1373
23.2	0.9138	7.610	0.1314	30.2	0.8739	7.277	0.1374
23.3	0.9132	7.605	0.1315	30.3	0.8734	7.273	0.1375
23.4	0.9126	7.600	0.1316	30.4	0.8728	7.268	0.1376
23.5	0.9121	7.595	0.1317	30.5	0.8723	7.264	0.1377
23.6	0.9115	7.590	0.1318	30.6	0.8717	7.259	0.1378
23.7	0.9109	7.585	0.1318	30.7	0.8712	7.254	0.1379
23.8	0.9103	7.580	0.1319	30.8	0.8706	7.249	0.1379
23.9	0.9097	7.575	0.1320	30.9	0.8701	7.245	0.1380
24.0	0.9091	7.570	0.1321	31.0	0.8696	7.241	0.1381
24.1	0.9085	7.565	0.1322	31.1	0.8690	7.236	0.1382
24.2	0.9079	7.561	0.1323	31.2	0.8685	7.232	0.1383
24.3	0.9073	7.556	0.1323	31.3	0.8679	7.227	0.1384
24.4	0.9067	7.551	0.1324	31.4	0.8674	7.223	001384
24.5	0.9061	7.546	0.1325	31.5	0.8669	7.218	0.1385
24.6	0.9056	7.541	0.1326	31.6	0.8663	7.214	0.1386
24.7	0.9050	7.536	0.1327	31.7	0.8658	7.210	0.1387
24.8	0.0944	7.531	0.1328	31.8	0.8653	7.205	0.1388
24.9	0.9038	7.526	0.1329	31.9	0.8647	7.201	0.1389
25.0	0.9032	7.522	0.1330	32.0	0.8642	7.196	0.1390
25.1	0.9026	7.517	0.1330	32.1	0.8637	7.192	0.1390
25.2	0.9021	7.512	0.1331	32.2	0.8631	7.187	0.1391
25.3	0.9015	7.507	0.1332	32.3	0.8626	7.183	0.1392
25.4	0.9009	7.502	0.1333	32.4	0.8621	7.178	0.1393
25.5	0.9003	7.497	0.1334	32.5	0.8615	7.173	0.1394
25.6	0.8997	7.493	0.1335	32.6	0.8610	7.169	0.1395
25.7	0.8992	7.588	0.1335	32.7	0.8605	7.165	0.1396
25.8	0.8986	7.488	0.1336	32.8	0.8600	7.161	0.1396
25.9	0.8980	7.478	0.1337	32.9	0.8594	7.156	0.1397
26.0	0.8974	7.473	0.1338	33.0	0.8589	7.152	0.1398
26.1	0.8969	7.469	0.1339	33.1	0.8584	7.147	0.1399
26.2	0.8963	7.464	0.1340	33.2	0.8578	7.143	0.1400
26.3	0.8957	7.459	0.1341	33.3	0.8573	7.139	0.1401
26.4	0.8951	7.454	0.1342	33.4	0.8568	7.134	0.1402
26.5	0.8946	7.449	0.1342	33.5	0.8563	7.130	0.1403
26.6	0.8940	7.445	0.1343	33.6	0.8557	7.125	0.1403
26.7	0.8934	7.440	0.1344	33.7	0.8552	7.121	0.1404
26.8	0.8929	7.435	0.1345	33.8	0.8547	7.117	0.1405
26.9	0.8923	7.430	0.1346	33.9	0.8542	7.113	0.1406
27.0	0.8917	7.425	0.1347	34.0	0.8537	7.108	0.1407
27.1	0.8912	7.421	0.1348	34.1	0.8531	7.104	0.1408
27.2	0.8906	7.416	0.1348	34.2	0.8526	7.100	0.1408
27.3	0.8900	7.411	0.1349	34.3	0.8521	7.095	0.1409
27.4	0.8895	7.407	0.1350	34.4	0.8516	7.091	0.1410
27.5	0.8889	7.402	0.1351	34.5	0.8511	7.087	0.1411
27.6	0.8883	7.397	0.1352	34.6	0.8505	7.082	0.1412
27.7	0.8878	7.393	0.1353	34.7	0.8500	7.078	0.1413
27.8	0.8872	7.388	0.1354	34.8	0.8495	7.074	0.1414
27.9	0.8866	7.383	0.1354	34.9	0.8490	7.069	0.1415
28.0	0.8861	7.378	0.1355	35.0	0.8485	7.065	0.1415
28.1	0.8855	7.374	0.1356	35.1	0.8480	7.061	0.1416
28.2	0.8850	7.369	0.1357	35.2	0.8475	7.057	0.1417
28.3	0.8844	7.365	0.1358	35.3	0.8469	7.052	0.1418
28.4	0.8838	7.360	0.1359	35.4	0.8464	7.048	0.1419
28.5	0.8833	7.355	0.1360	35.5	0.8459	7.044	0.1420
28.6	0.8827	7.351	0.1360	35.6	0.8454	7.039	0.1421
28.7	0.8822	7.346	0.1361	35.7	0.8449	7.035	0.1421
28.8	0.8816	7.341	0.1362	35.8	0.8444	7.031	0.1422
28.9	0.8811	7.337	0.1363	35.9	0.8439	7.027	0.1423

Degrees Baumé	Specific gravity at 60°/60°F.	Pounds per gallon	Gallons per pound	Degrees Baumé	Specific gravity at 60°/60°F.	Pounds per gallon	Gallons per pound
36.0	0.8434	7.022	0.1424	43.0	0.8092	6.738	0.1484
36.1	0.8429	7.018	0.1425	43.1	0.8088	6.734	0.1485
36.2	0.8424	7.014	0.1426	43.2	0.8083	6.730	0.1486
36.3	0.8419	7.010	0.1427	43.3	0.8078	6.726	0.1487
36.4	0.8413	7.006	0.1427	43.4	0.8074	6.722	0.1488
36.5	0.8408	7.001	0.1428	43.5	0.8069	6.718	0.1489
36.6	0.8403	6.997	0.1429	43.6	0.8065	6.715	0.1489
36.7	0.8398	6.993	0.1430	43.7	0.8060	6.711	0.1490
36.8	0.8393	6.989	0.1431	43.8	0.8055	6.707	0.1491
36.9	0.8388	6.985	0.1432	43.9	0.8051	6.703	0.1492
37.0	0.8383	6.980	0.1433	44.0	0.8046	6.699	0.1493
37.1	0.8378	6.976	0.1433	44.1	0.8041	6.695	0.1494
37.2	0.8373	6.972	0.1434	44.2	0.8037	6.691	0.1495
37.3	0.8368	6.968	0.1435	44.3	0.8032	6.688	0.1495
37.4	0.8363	6.964	0.1436	44.4	0.8028	6.684	0.1496
37.5	0.8358	6.960	0.1437	44.5	0.8023	6.680	0.1497
37.6	0.8353	6.955	0.1438	44.6	0.8018	6.676	0.1498
37.7	0.8348	6.951	0.1439	44.7	0.8014	6.672	0.1499
37.8	0.8343	6.947	0.1439	44.8	0.8009	6.668	0.1500
37.9	0.8338	6.943	0.1440	44.9	0.8005	6.665	0.1500
38.0	0.8333	6.939	0.1441	45.0	0.8000	6.661	0.1501
38.1	0.8328	6.935	0.1442	45.1	0.7995	6.657	0.1502
38.2	0.8323	6.930	0.1443	45.2	0.7991	6.653	0.1503
38.3	0.8318	6.926	0.1444	45.3	0.7986	6.649	0.1504
38.4	0.8314	6.922	0.1445	45.4	0.7982	6.646	0.1505
38.5	0.8309	6.918	0.1446	45.5	0.7977	6.642	0.1506
38.6	0.8304	6.914	0.1446	45.6	0.7973	6.638	0.1506
38.7	0.8299	6.910	0.1447	45.7	0.7968	6.634	0.1507
38.8	0.8294	6.906	0.1448	45.8	0.7964	6.630	0.1508
38.9	0.8289	6.902	0.1449	45.9	0.7959	6.627	0.1509
39.0	0.8284	6.898	0.1450	46.0	0.7955	6.623	0.1510
39.1	0.8279	6.894	0.1451	46.1	0.7950	6.619	0.1511
39.2	0.8274	6.889	0.1452	46.2	0.7946	6.615	0.1512
39.3	0.8269	6.885	0.1452	46.3	0.7941	6.612	0.1512
39.4	0.8264	6.881	0.1453	46.4	0.7937	6.608	0.1513
39.5	0.8260	6.877	0.1454	46.5	0.7932	6.604	0.1514
39.6	0.8255	6.873	0.1455	46.6	0.7928	6.600	0.1515
39.7	0.8250	6.869	0.1456	46.7	0.7923	6.597	0.1516
39.8	0.8245	6.865	0.1457	46.8	0.7919	6.593	0.1517
39.9	0.8240	6.861	0.1458	46.9	0.7914	6.589	0.1518
40.0	0.8235	6.857	0.1459	47.0	0.7910	6.586	0.1518
40.1	0.8230	6.853	0.1459	47.1	0.7905	6.582	0.1519
40.2	0.8226	6.849	0.1460	47.2	0.7901	6.578	0.1520
40.3	0.8221	6.845	0.1461	47.3	0.7896	6.574	0.1521
40.4	0.8216	6.841	0.1462	47.4	0.7892	6.571	0.1522
40.5	0.8211	6.837	0.1463	47.5	0.7887	6.567	0.1523
40.6	0.8206	6.833	0.1463	47.6	0.7883	6.563	0.1524
40.7	0.8202	6.829	0.1464	47.7	0.7878	6.560	0.1524
40.8	0.8197	6.825	0.1465	47.8	0.7874	6.556	0.1525
40.9	0.8192	6.821	0.1466	47.9	0.7870	6.552	0.1526
41.0	0.8187	6.817	0.1467	48.0	0.7865	6.548	0.1527
41.1	0.8182	6.813	0.1468	48.1	0.7861	6.545	0.1528
41.2	0.8178	6.809	0.1469	48.2	0.7856	6.541	0.1529
41.3	0.8173	6.805	0.1470	48.3	0.7852	6.537	0.1530
41.4	0.8168	6.801	0.1470	48.4	0.7848	6.534	0.1530
41.5	0.8163	6.797	0.1471	48.5	0.7843	6.530	0.1531
41.6	0.8159	6.793	0.1472	48.6	0.7839	6.526	0.1532
41.7	0.8154	6.789	0.1473	48.7	0.7834	6.523	0.1533
41.8	0.8149	6.785	0.1474	48.8	0.7830	6.519	0.1534
41.9	0.8144	6.781	0.1475	48.9	0.7826	6.515	0.1535
42.0	0.8140	6.777	0.1476	49.0	0.7821	6.511	0.1536
42.1	0.8135	6.773	0.1476	49.1	0.7817	6.508	0.1537
42.2	0.8130	6.769	0.1477	49.2	0.7812	6.504	0.1538
42.3	0.8125	6.765	0.1478	49.3	0.7808	6.501	0.1538
42.4	0.8121	6.761	0.1479	49.4	0.7804	6.497	0.1539
42.5	0.8116	6.758	0.1480	49.5	0.7799	6.494	0.1540
42.6	0.8111	6.754	0.1481	49.6	0.7795	6.490	0.1541
42.7	0.8107	6.750	0.1481	49.7	0.7791	6.486	0.1542
42.8	0.8102	6.746	0.1482	49.8	0.7786	6.483	0.1542
42.9	0.8097	6.742	0.1483	49.9	0.7782	6.479	0.1543

Degrees Baumé	Specific gravity at 60°/60°F.	Pounds per gallon	Gallons per pound	Degrees Baumé	Specific gravity at 60°/60°F.	Pounds per gallon	Gallons per pound
50.0	0.7778	6.476	0.1544	57.0	0.7487	6.233	0.1604
50.1	0.7773	6.472	0.1545	57.1	0.7483	6.229	0.1605
50.2	0.7769	6.468	0.1546	57.2	0.7479	6.226	0.1606
50.3	0.7765	6.465	0.1547	57.3	0.7475	6.223	0.1607
50.4	0.7761	6.461	0.1548	57.4	0.7471	6.219	0.1608
50.5	0.7756	6.458	0.1548	57.5	0.7467	6.216	0.1609
50.6	0.7752	6.454	0.1549	57.6	0.7463	6.213	0.1610
50.7	0.7748	6.450	0.1550	57.7	0.7459	6.209	0.1611
50.8	0.7743	6.447	0.1551	57.8	0.7455	6.206	0.1611
50.9	0.7739	6.443	0.1552	57.9	0.7451	6.203	0.1612
51.0	0.7735	6.440	0.1553	58.0	0.7447	6.199	0.1613
51.1	0.7731	6.436	0.1554	58.1	0.7443	6.196	0.1614
51.2	0.7726	6.432	0.1555	58.2	0.7439	6.193	0.1615
51.3	0.7722	6.429	0.1555	58.3	0.7435	6.190	0.1616
51.4	0.7718	6.425	0.1556	58.4	0.7431	6.186	0.1617
15.5	0.7713	6.421	0.1557	58.5	0.7427	6.183	0.1617
51.6	0.7709	6.418	0.1558	58.6	0.7423	6.180	0.1618
51.7	0.7705	6.415	0.1559	58.7	0.7419	6.176	0.1619
51.8	0.7701	6.411	0.1560	58.8	0.7415	6.173	0.1620
51.9	0.7697	6.408	0.1561	58.9	0.7411	6.170	0.1621
52.0	0.7692	6.404	0.1562	59.0	0.7407	6.166	0.1622
52.1	0.7688	6.401	0.1562	59.1	0.7403	6.163	0.1623
52.2	0.7684	6.397	0.1563	59.2	0.7400	6.160	0.1623
52.3	0.7680	6.394	0.1564	59.3	0.7396	6.157	0.1624
52.4	0.7675	6.390	0.1565	59.4	0.7392	6.154	0.1625
25.5	0.7671	6.387	0.1566	59.5	0.7388	6.150	0.1626
52.6	0.7667	6.383	0.1567	59.6	0.7384	6.147	0.1627
52.7	0.7663	6.380	0.1567	59.7	0.7380	6.144	0.1628
52.8	0.7659	6.376	0.1568	59.8	0.7376	6.141	0.1628
52.9	0.7654	6.373	0.1569	59.9	0.7372	6.137	0.1629
53.0	0.7650	6.369	0.1570	60.0	0.7368	6.134	0.1630
53.1	0.7646	6.366	0.1571	60.1	0.7365	6.131	0.1631
53.2	0.7642	3.362	0.1572	60.2	0.7361	6.128	0.1632
53.3	0.7638	6.359	0.1573	60.3	0.7357	6.124	0.1633
53.4	0.7634	6.355	0.1574	60.4	0.7353	6.121	0.1634
53.5	0.7629	6.351	0.1574	60.5	0.7349	6.118	0.1635
53.6	0.7625	6.348	0.1575	60.6	0.7345	6.115	0.1635
53.7	0.7621	6.345	0.1576	60.7	0.7341	6.112	0.1636
53.8	0.7617	6.341	0.1577	60.8	0.7338	6.108	0.1637
53.9	0.7613	6.338	0.1578	60.9	0.7334	6.105	0.1638
54.0	0.7609	6.334	0.1579	61.0	0.7330	6.102	0.1639
54.1	0 7605	6.331	0.1580	61.1	0.7326	6.099	0.1640
54.2	0.7600	6.327	0.1581	61.2	0.7322	6.096	0.1640
54.3	0.7596	6.324	0.1581	61.3	0.7318	6.093	0.1641
54.4	0.7592	6.321	0.1582	61.4	0.7315	6.090	0.1642
54.5	0.7588	6.317	0.1583	61.5	0.7311	6.086	0.1643
54.6	0.7584	6.314	0.1584	61.6	0.7307	6.083	0.1644
54.7	0.7580	6.311	0.1585	61.7	0.7303	6.080	0.1645
54.8	0.7576	6.307	0.1586	61.8	0.7299	6.077	0.1646
54.9	0.7572	6.304	0.1586	61.9	0.7295	6.073	0.1647
55.0	0.7568	6.300	0.1587	62.0	0.7292	6.070	0.1647
55.1	0.7563	6.296	0.1588	62.1	0.7288	6.067	0.1648
55.2	0.7559	6.293	0.1589	62.2	0.7284	6.064	0.1649
55.3	0.7555	6.290	0.1590	62.3	0.7280	6.060	0.1650
55.4	0.7551	6.287	0.1591	62.4	0.7277	6.057	0.1651
55.5	0.7547	6.283	0.1592	62.5	0.7273	6.054	0.1652
55.6	0.7543	6.280	0.1592	62.6	0.7269	6.051	0.1653
55.7	0.7539	6.276	0.1593	62.7	0.7265	6.048	0.1653
55.8	0.7535	6.273	0.1594	62.8	0.7261	6.045	0.1654
55.9	0.7531	6.270	0.1595	62.9	0.7258	6.042	0.1655
56.0	0.7527	6.266	0.1596	63.0	0.7254	6.038	0.1656
56.1	0.7523	6.263	0.1597	63.1	0.7250	6.035	0.1657
56.2	0.7519	6.259	0.1598	63.2	0.7246	6.032	0.1658
56.3	0.7515	6.256	0.1598	63.3	0.7243	6.029	0.1659
56.4	0.7511	6.253	0.1599	63.4	0.7239	6.026	0.1659
56.5	0.7507	6.249	0.1600	63.5	0.7235	6.023	0.1660
56.6	0.7503	6.246	0.1601	63.6	0.6231	6.020	0.1661
56.7	0.7499	6.243	0.1602	63.7	0.7228	6.017	0.1662
56.8	0.7495	6.240	0.1603	63.8	0.7224	6.014	0.1663
56.9	0.7491	6.236	0.1604	63.9	0.7220	6.010	0.1664

Degrees Baumé	Specific gravity at 60°/60°F.	Pounds per gallon	Gallons per pound	Degrees Baumé	Specific gravity at 60°/60°F.	Pounds per gallon	Gallons per pound
64.0	0.7216	6.007	0.1665	71.0	0.6965	5.798	0.1725
64.1	0.7213	6.004	0.1666	71.1	0.6962	5.795	0.1726
64.2	0.7209	6.001	0.1666	71.2	0.6958	5.792	0.1727
64.3	0.7205	5.998	0.1667	71.3	0.6955	5 789	0.1727
64.4	0.7202	5.995	0.1668	71.4	0.6951	5.786	0.1728
64.5	0.7198	5.992	0.1669	71.5	0.6948	5.784	0.1729
64.6	0.7194	5.989	0.1670	71.6	0.6944	5.781	0.1730
64.7	0.7191	5.986	0.1671	71.7	0.6941	5.778	0.1731
64.8	0.7187	5.983	0.1671	71.8	0.6938	5.775	0.1732
64.9	0.7183	5.980	0.1672	71.9	0.6934	5.772	0.1733
65.0	0.7179	5.976	0.1673	72.0	0.6931	5.769	0.1733
65.1	0.7176	5.973	0.1674	72.1	0.6927	5.766	0.1734
65.2	0.7172	5.970	0.1675	72.2	0.6924	5.763	0.1735
65.3	0.7168	5.967	0.1676	72.3	0.6920	5.760	0.1736
65.4	0.7165	5.964	0.1677	72.4	0.6917	5.758	0.1737
65.5	0.7161	5.961	0.1678	72.5	0.6914	5.755	0.1738
65.6	0.7157	5.958	0.1678	72.6	0.6910	5.752	0.1739
65.7	0.7154	5.955	0.1679	72.7	0.6907	5.749	0.1739
65.8	0.7150	5.952	0.1680	72.8	0.6903	5.746	0.1740
65.9	0.7147	5.949	0.1681	72.9	0.6900	5.744	0.1741
66.0	0.7143	5.946	0.1682	73.0	0.6897	5.741	0.1742
66.1	0.7139	5.943	0.1683	73.1	0.6893	5.738	0.1743
66.2	0.7136	5.940	0.1684	73.2	0.6890	5.735	0.1744
66.3	0.7132	5.937	0.1684	73.3	0.6886	5.732	0.1745
66.4	0.7128	5.934	0.1685	73.4	0.6883	5.729	0.1746
66.5	0.7125	5.931	0.1686	73.5	0.6880	5.727	0.1746
66.6	0.7121	5.928	0.1687	73.6	0.6876	5.724	0.1747
66.7	0.7117	5.925	0.1688	73.7	0.6873	5.721	0.1748
66.8	0.7114	5.922	0.1689	73.8	0.6869	5.718	0.1749
66.9	0.7110	5.919	0.1689	73.9	0.6866	5.715	0.1750
67.0	0.7107	5.916	0.1690	74.0	0.6863	5.712	0.1751
67.1	0.7103	5.913	0.1591	74.1	0.6859	5.710	0.1751
67.2	0.7099	5.910	0.1692	74.2	0.6856	5.707	0.1752
67.3	0.7096	5.907	0.1693	74.3	0.6853	5.704	0.1753
67.4	0.7092	5.904	0.1694	74.4	0.6849	5.701	0.1754
67.5	0.7089	5.901	0.1695	74.5	0.6846	5 698	0.1755
67.6	0.7085	5.898	0.1695	74.6	0.6843	5.696	0.1756
67.7	0.7081	5.895	0.1696	74.7	0.6839	5.693	0.1757
67.8	0.7078	5.892	0.1697	74.8	0.6836	5.690	0.1757
67.9	0.7074	5.889	0.1698	74.9	0.6833	5.687	0.1758
68.0	0.7071	5.886	0.1699	75.0	0.6829	5.685	0.1759
68.1	0.7067	5.883	0.1700	75.1	0.6826	5.682	0.1760
68.2	0.7064	5.880	0.1701	75.2	0.6823	5.679	0.1761
68.3	0.7060	5.877	0.1702	75.3	0.6819	5.676	0.1762
68.4	0.7056	5.874	0.1702	75.4	0.6816	5.673	0.1763
68.5	0.7053	5.871	0.1703	75.5	0.6813	5.671	0.1763
68.6	0.7049	5.868	0.1704	75.6	0.6809	5.668	0.1764
68.7	0.7046	5.865	0.1705	75.7	0.6806	5.665	0.1765
68.8	0.7042	5.862	0.1706	75.8	0.6803	5.662	0.1766
68.9	0.7039	5.859	0.1707	75.9	0.6799	5.660	0.1767
69.0	0.7035	5.856	0.1708	76.0	0.6796	5.657	0.1768
69.1	0.7032	5.853	0.1709	76.1	0.6793	5.654	0.1769
69.2	0.7028	5.850	0.1709	76.2	0.6790	5.652	0.1769
69.3	0.7025	5.848	0.1710	76.3	0.6786	5.649	0.1770
69.4	0.7021	5.845	0.1711	76.4	0.6783	5.646	0.1771
69.5	0.7018	5.842	0.1712	76.5	0.6780	5.643	0.1772
69.6	0.7014	5.839	0.1713	76.6	0.6776	5.640	0.1773
69.7	0.7011	5.836	0.1714	76.7	0.6773	5.638	0.1774
69.8	0.7007	5.833	0.1714	76.8	0.6770	5.635	0.1775
69.9	0.7004	5.830	0.1715	76.9	0.6767	5.632	0.1776
70.0	0.7000	5.827	0.1716	77.0	0.6763	5.629	0.1776
70.1	0.6997	5.824	0.1717	77.1	0.6760	5.627	0.1777
70.2	0.6993	5.821	0.1718	77.2	0.6757	5.624	0.1778
70.3	0.6990	5.818	0.1719	77.3	0.6753	5.621	0.1779
70.4	0.6986	5.815	0.1720	77.4	0.6750	5.618	0.1780
70.5	0.6983	5.812	0.1721	77.5	0.6747	5.616	0.1781
70.6	0.6979	5.810	0.1721	77.6	0.6744	5.613	0.1782
70.7	0.6976	5.807	0.1722	77.7	0.6740	5.610	0.1783
70.8	0.6972	5.804	0.1723	77.8	0.6737	5.608	0.1783
70.9	0.6969	5.801	0.1724	77.9	0.6734	5.605	0.1784

Degrees Baumé	Specific gravity at 60°/60°F.	Pounds per gallon	Gallons per pound	Degrees Baumé	Specific gravity at 60°/60°F.	Pounds per gallon	Gallons per pound
78.0	0.6731	5.602	0.1785	85.0	0.6512	5.420	0.1845
78.1	0.6728	5.600	0.1786	85.1	0.6509	5.417	0.1846
78.2	0.6724	5.597	0.1787	85.2	0.6506	5.415	0.1847
78.3	0.6721	5.594	0.1788	85.3	0.6503	5.412	0.1848
78.4	0.6718	5.592	0.1788	85.4	0.6500	5.410	0.1848
78.5	0.6715	5.589	0.1789	85.5	0.6497	5.407	0.1849
78.6	0.6711	5.586	0.1790	85.6	0.6494	5.405	0.1850
78.7	0.6708	5.584	0.1791	85.7	0.6490	5.402	0.1851
78.8	0.6705	5.581	0.1792	85.8	0.6487	5.400	0.1852
78.9	0.6702	5.578	0.1793	85.9	0.6484	5.397	0.1853
79.0	0.6699	5.576	0.1793	86.0	0.6482	5.395	0.1854
79.1	0.6695	5.573	0.1794	86.1	0.6479	5.392	0.1855
79.2	0.6692	5.570	0.1795	86.2	0.6476	5.390	0.1855
79.3	0.6689	5.568	0.1796	86.3	0.6473	5.387	0.1856
79.4	0.6686	5.565	0.1797	86.4	0.6470	5.385	0.1857
79.5	0.6683	5.562	0.1798	86.5	0.6467	5.382	0.1858
79.6	0.6679	5.560	0.1799	86.6	0.6464	5.380	0.1859
79.7	0.6676	5.557	0.1800	86.7	0.6461	5.377	0.1860
79.8	0.6673	5.554	0.1801	86.8	0.6458	5.375	0.1860
79.9	0.6670	5.552	0.1801	86.9	0.6455	5.372	0.1861
80.0	0.6667	5.549	0.1802	87.0	0.6452	5.370	0.1862
80.1	0.6663	5.546	0.1803	87.1	0.6449	5.367	0.1863
80.2	0.6660	5.543	0.1804	87.2	0.6446	5.365	0.1864
80.3	0.6657	5.541	0.1805	87.3	0.6443	5.362	0.1865
80.4	0.6654	5.538	0.1806	87.4	0.6440	5.360	0.1866
80.5	0.6651	5.536	0.1806	87.5	0.6437	5.357	0.1867
80.6	0.6648	5.533	0.1807	87.6	0.6434	5.355	0.1867
80.7	0.6645	5.531	0.1808	87.7	0.6431	5.352	0.1868
80.8	0.6641	5.528	0.1809	87.8	0.6428	5.350	0.1869
80.9	0.6638	5.525	0.1810	87.9	0.6425	5.347	0.1870
81.0	0.6635	5.522	0.1811	88.0	0.6422	5.345	0.1871
81.1	0.6632	5.520	0.1812	88.1	0.6419	5.343	0.1872
81.2	0.6629	5.517	0.1813	88.2	0.6416	5.340	0.1873
81.3	0.6626	5.515	0.1813	88.3	0.6413	5.338	0.1873
81.4	0.6623	5.512	0.1814	88.4	0.6410	5.335	0.1874
81.5	0.6619	5.510	0.1815	88.5	0.6407	5.333	0.1875
81.6	0.6616	5.507	0.1816	88.6	0.6404	5.330	0.1876
81.7	0.6613	5.504	0.1817	88.7	0.6401	5.328	0.1877
81.8	0.6610	5.502	0.1818	88.8	0.6399	5.325	0.1878
81.9	0.6607	5.499	0.1819	88.9	0.6396	5.323	0.1879
82.0	0.6604	5.497	0.1819	89.0	0.6393	5.320	0.1880
82.1	0.6601	5.494	0.1820	89.1	0.6390	5.318	0.1880
82.2	0.6598	5.491	0.1821	89.2	0.6387	5.316	0.1881
82.3	0.6594	5.489	0.1822	89.3	0.6384	5.313	0.1882
82.4	0.6591	5.486	0.1823	89.4	0.6381	5.311	0.1883
82.5	0.6588	5.484	0.1823	89.5	0.6378	5.308	0.1884
82.6	0.6585	5.481	0.1824	89.6	0.6375	5.306	0.1885
82.7	0.6582	5.478	0.1825	89.7	0.6372	5.304	0.1885
82.8	0.6579	5.476	0.1826	89.8	0.6369	5.301	0.1886
82.9	0.6576	5.473	0.1827	89.9	0.6367	5.299	0.1887
83.0	0.6573	5.471	0.1828	90.0	0.6364	5.296	0.1888
83.1	0.6570	5.468	0.1829	90.1	0.6361	5.294	0.1889
83.2	0.6567	5.466	0.1829	90.2	0.6358	5.291	0.1890
83.3	0.6564	5.463	0.1830	90.3	0.6355	5.289	0.1891
83.4	0.6560	5.460	0.1831	90.4	0.6352	5.286	0.1892
83.5	0.6557	5.458	0.1832	90.5	0.6349	5.284	0.1893
83.6	0.6554	5.455	0.1833	90.6	0.6346	5.281	0.1894
83.7	0.6551	5.453	0.1834	90.7	0.6343	5.279	0.1894
83.8	0.6548	5.450	0.1835	90.8	0.6341	5.277	0.1895
83.9	0.6545	5.448	0.1836	90.9	0.6338	5.275	0.1896
84.0	0.6542	5.445	0.1837	91.0	0.6335	5.272	0.1897
84.1	0.6539	5.443	0.1837	91.1	0.6332	5.270	0.1898
84.2	0.6536	5.440	0.1838	91.2	0.6329	5.267	0.1899
84.3	0.6533	5.437	0.1839	91.3	0.6326	5.265	0.1899
84.4	0.6530	5.435	0.1840	91.4	0.6323	5.263	0.1900
84.5	0.6527	5.432	0.1841	91.5	0.6321	5.261	0.1901
84.6	0.6524	5.430	0.1842	91.6	0.6318	5.258	0.1902
84.7	0.6521	5.427	0.1843	91.7	0.6315	5.256	0.1903
84.8	0.6518	5.425	0.1843	91.8	0.6312	5.253	0.1904
84.9	0.6515	5.422	0.1844	91.9	0.6309	5.251	0.1904

Degrees Baumé	Specific gravity at 60°/60°F.	Pounds per gallon	Gallons per pound	Degrees Baumé	Specific gravity at 60°/60°F.	Pounds per gallon	Gallons per pound
92.0	0.6306	5.248	0.1905	96.0	0.6195	5.155	0.1940
92.1	0.6303	5.246	0.1906	96.1	0.6192	5.153	0.1941
92.2	0.6301	5.244	0.1907	96.2	0.6189	5.150	0.1942
92.3	0.6298	5.241	0.1908	96.3	0.6186	5.148	0.1943
92.4	0.6295	5.239	0.1909	96.4	0.6184	5.146	0.1943
92.5	0.6292	5.236	0.1910	96.5	0.6181	5.144	0.1944
92.6	0.6289	5.234	0.1911	96.6	0.6178	5.142	0.1945
92.7	0.6286	5.232	0.1911	96.7	0.6176	5.140	0.1946
92.8	0.6284	5.230	0.1912	96.8	0.6173	5.137	0.1947
92.9	0.6281	5.227	0.1913	96.9	0.6170	5.135	0.1948
93.0	0.6278	5.225	0.1914	97.0	0.6167	5.132	0.1949
93.1	0.6275	5.222	0.1915	97.1	0.6165	5.130	0.1949
93.2	0.6272	5.220	0.1916	97.2	0.6162	5.128	0.1950
93.3	0.6270	5.218	0.1916	97.3	0.6159	5.126	0.1951
93.4	0.6267	5.216	0.1917	97.4	0.6157	5.124	0.1952
93.5	0.6264	5.213	0.1918	97.5	0.6154	5.121	0.1953
93.6	0.6261	5.210	0.1919	97.6	0.6151	5.119	0.1954
93.7	0.6258	5.208	0.1920	97.7	0.6148	5.116	0.1955
93.8	0.6256	5.206	0.1921	97.8	0.6146	5.114	0.1955
93.9	0.6253	5.204	0.1922	97.9	0.6143	5.112	0.1956
94.0	0.6250	5.201	0.1923	98.0	0.6140	5.110	0.1957
94.1	0.6247	5.199	0.1924	98.1	0.6138	5.108	0.1958
94.2	0.6244	5.196	0.1925	98.2	0.6135	5.106	0.1959
94.3	0.6242	5.194	0.1925	98.3	0.6132	5.103	0.1960
94.4	0.6239	5.192	0.1926	98.4	0.6130	5.101	0.1960
94.5	0.6236	5.190	0.1927	98.5	0.6127	5.099	0.1961
94.6	0.6233	5.187	0.1928	98.6	0.6124	5.096	0.1962
94.7	0.6231	5.185	0.1929	98.7	0.6122	5.094	0.1963
94.8	0.6228	5.183	0.1929	98.8	0.6119	5.092	0.1964
94.9	0.6225	5.180	0.1930	98.9	0.6116	5.090	0.1965
95.0	0.6222	5.178	0.1931	99.0	0.6114	5.088	0.1966
95.1	0.6219	5.176	0.1932	99.1	0.6111	5.085	0.1967
95.2	0.6217	5.174	0.1933	99.2	0.6108	5.083	0.1967
95.3	0.6214	5.171	0.1934	99.3	0.6106	5.081	0.1968
95.4	0.6211	5.169	0.1935	99.4	0.6103	5.079	0.1969
95.5	0.6208	5.166	0.1936	99.5	0.6100	5.076	0.1970
95.6	0.6206	5.164	0.1936	99.6	0.6098	5.074	0.1971
95.7	0.6203	5.162	0.1937	99.7	0.6095	5.072	0.1972
95.8	0.6200	5.160	0.1938	99.8	0.6092	5.070	0.1972
95.9	0.6197	5.157	0.1939	99.9	0.6090	5.068	0.1973
				100.0	0.6087	5.066	0.1974

The Properties of Some of the Paraffin Hydrocarbons (Liquid and Solid Under Ordinary Conditions) Found in Petroleum

Hydrocarbon	Molecular formula	Bé. gravity	B.p. °C.	B.p. °F.	M.p. °C.	Vapor in cu. ft. from 1 gal.
Liquid:						
Pentane	C_5H_{12}	94.0	36.3	97	.....	31
Hexane	C_6H_{14}	81.0	69.0	156	.....	27
Heptane	C_7H_{16}	73.0	98 4	208	.....	25
Octane	C_8H_{18}	70.0	125.5	258	.....	
Nonane	C_9H_{20}	65.0	149.5	301	−51	
Decane	$C_{10}H_{22}$	61.0	173.0	343	−31	
Undecane	$C_{11}H_{24}$	51.0	195.0	383	−26	
Dodecane	$C_{12}H_{26}$	51.0	214.0	417	−12	
Tridecane	$C_{13}H_{28}$	51.0	234.0	453	− 6	
Tetradecane	$C_{14}H_{30}$	50.5	252.0	486	+ 5	
Pentadecane	$C_{15}H_{32}$	50.0	270.0	518	10	
Hexadecane	$C_{16}H_{34}$	50.0	287.0	549	18	
Solid:						
Octadecane	$C_{18}H_{38}$	....	317.0	602	28	
Eicosane	$C_{20}H_{42}$	....	.....	...	37	
Tricosane	$C_{23}H_{48}$	....	.....	...	48	
Tetracosane	$C_{24}H_{50}$	....	.....	...	51	
Pentacosane	$C_{25}H_{52}$	....	.....	...	53–54	
Hexacosane	$C_{26}H_{54}$	....	.....	...	55–56	
Octocosane	$C_{28}H_{58}$	....	.....	...	60	
Nonocosane	$C_{29}H_{60}$	....	.....	...	62–63	
Hentriacontane	$C_{31}H_{64}$	....	.....	...	68	
Dotriacontane	$C_{32}H_{66}$	....	.....	...	70	
Tetratriacontane	$C_{34}H_{70}$	....	.....	...	71–72	
Pentatriacontane	$C_{35}H_{72}$	....	.....	...	75	

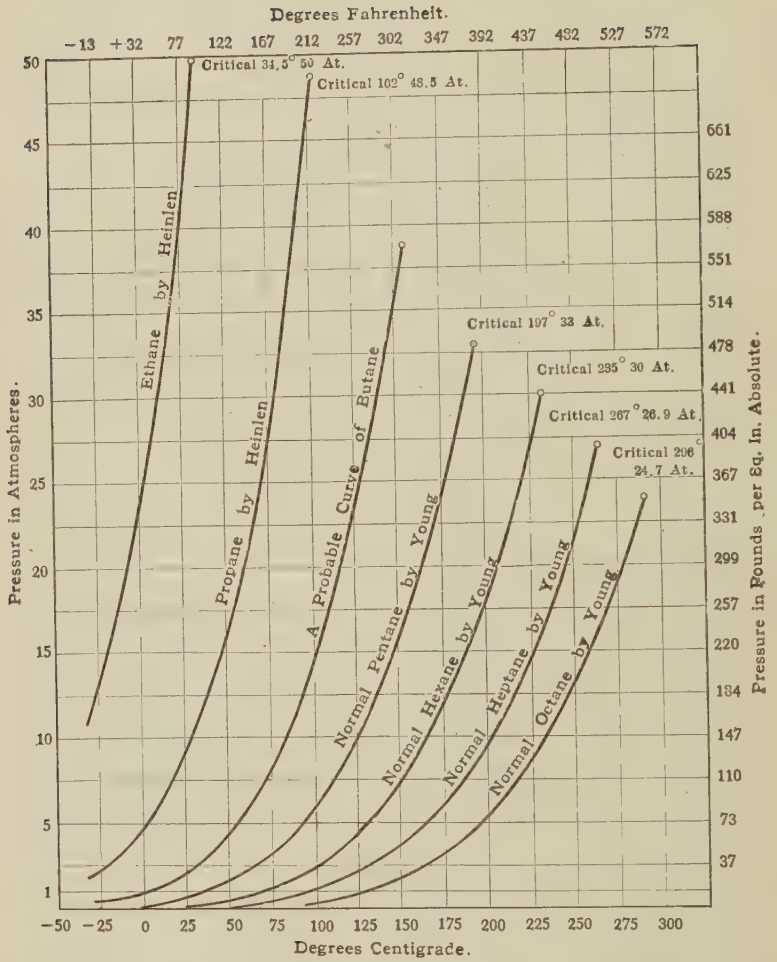

Degrees Fahrenheit.
−13 +32 77 122 167 212 257 302 347 392 437 482 527 572
Critical 34.5° 50 At.
Critical 102° 48.5 At.
Critical 197° 33 At.
Critical 235° 30 At.
Critical 267° 26.9 At.
Critical 296° 24.7 At.
Ethane by Heinlen
Propane by Heinlen
A Probable Curve of Butane
Normal Pentane by Young
Normal Hexane by Young
Normal Heptane by Young
Normal Octane by Young
Pressure in Atmospheres.
Pressure in Pounds per Sq. In. Absolute.
50 45 40 35 30 25 20 15 10 5 1
661 625 588 551 514 478 441 404 367 331 299 257 220 184 147 110 73 37
−50 −25 0 25 50 75 100 125 150 175 200 225 250 275 300
Degrees Centigrade.

FIG. 110.—Vapor tension curves of hydrocarbons of the paraffin series. (Data collected by W. O. Snelling.)

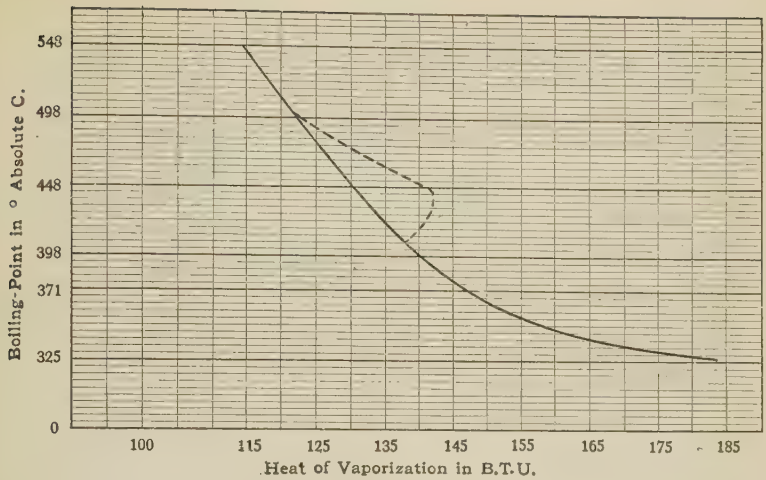


FIG. 111.—Curve presenting the variation of heat of vaporization with boiling-point. (Data from Rittman, arranged graphically by O. W. Bassett.)

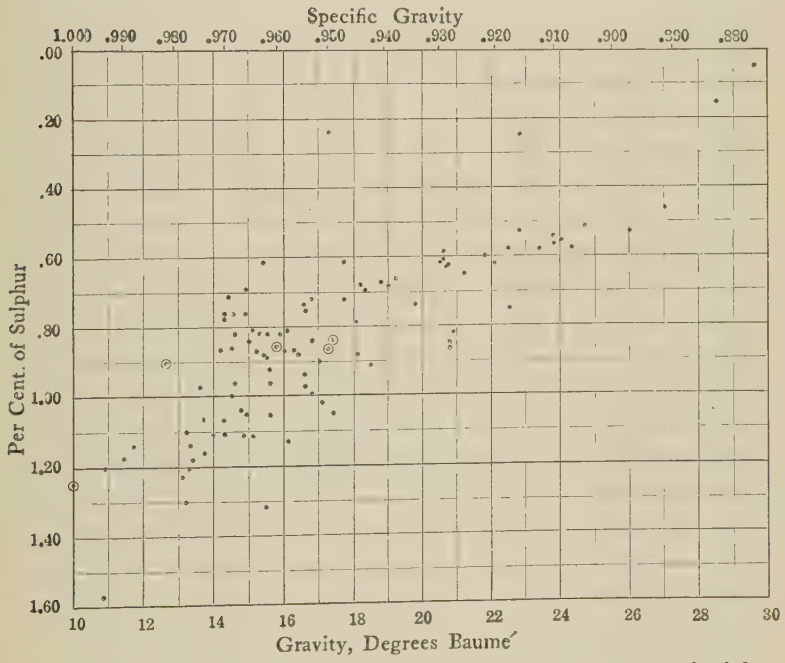


FIG. 112.—Diagram showing the relation of gravity to percentage of sulphur in 100 samples of oil from the Sunset-Midway field. Analyses represented by small solid circles made by Bureau of Mines; the others by different chemists.

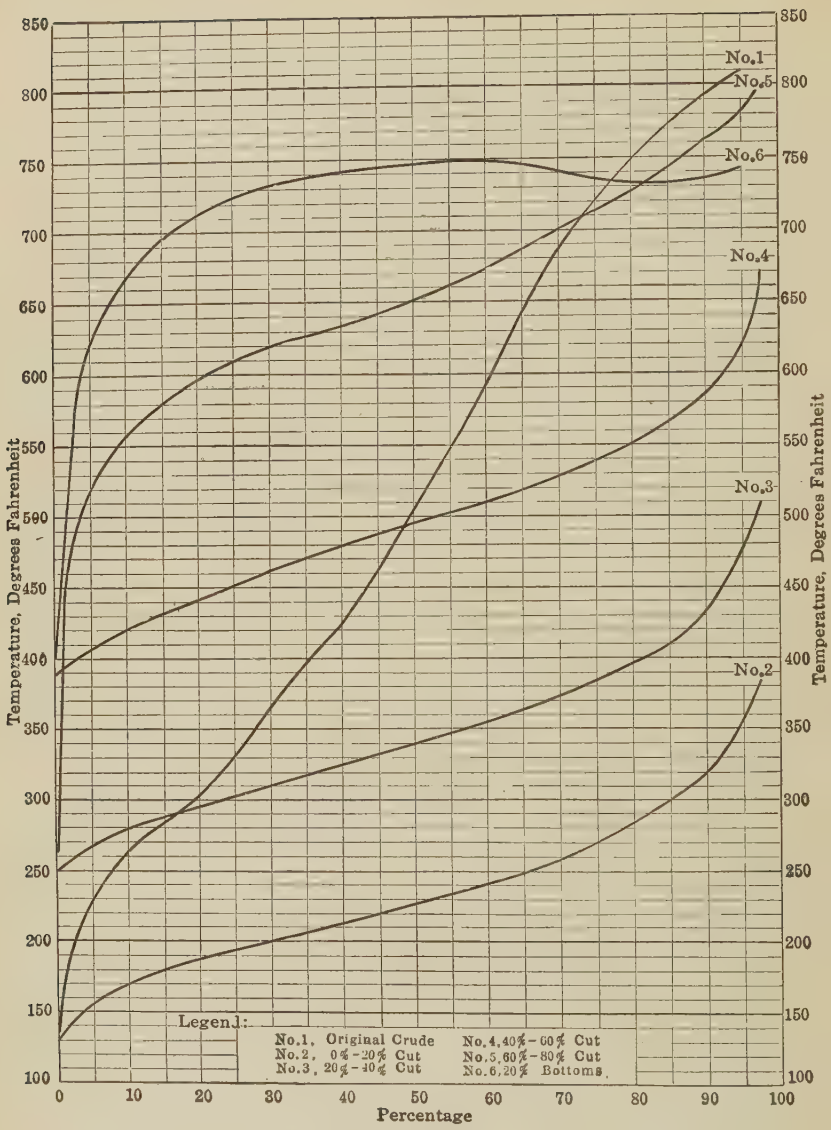


FIG. 113.—Distillation curves on Cushing crude petroleum. (W. E. Espy.)

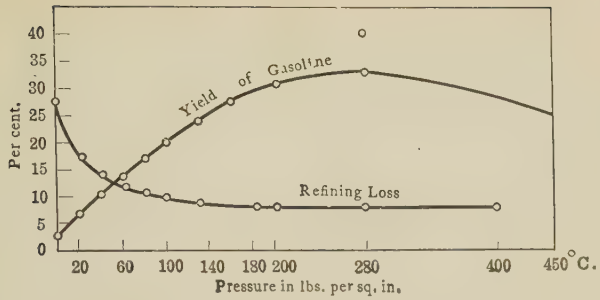


FIG. 114.—Curves showing the variation of gasoline yield and loss to sulphuric acid in the pressure distillation of reduced oil.

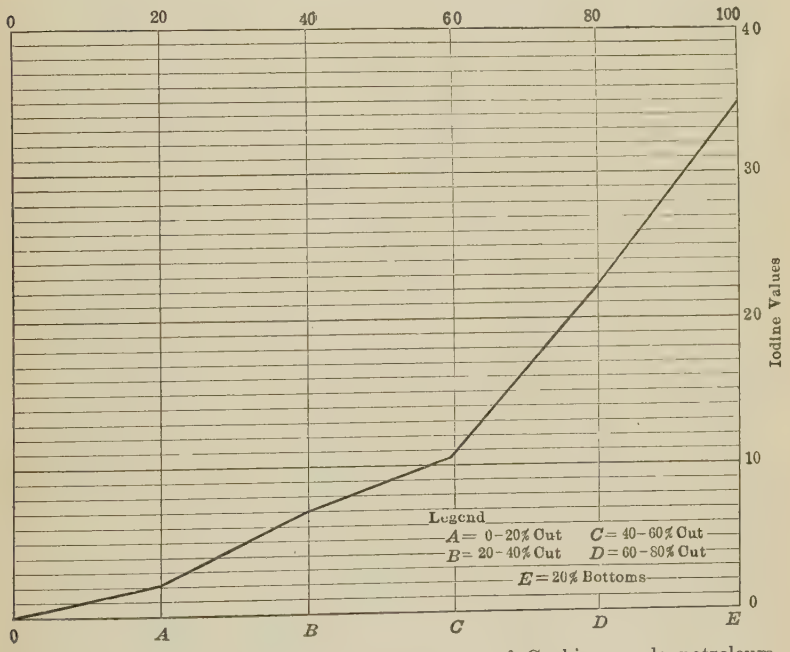


FIG. 115.—Iodine values on 20 per cent. cuts of Cushing crude petroleum. (W. E. Espy.)

The Products of the Distillation of Mid-Continent Crude Petroleums

The accompanying table, giving the analysis of twenty-seven of the principal Mid-Continent crudes, with tests of their products, probably furnishes the most complete data on the largest number of crudes ever published. The table was prepared in the laboratories of Cosden & Company at West Tulsa, under the direction of Charles K. Francis.

In each case the gravity, initial boiling-point and B.S. content of the crude are shown, together with its yield of benzine, naphtha and bottoms.

Crude	Analysis						
Showing State and District	Grav., °Bé.	I.B.P., °F.	Per cent. B.S.	Per cent. benz.	Per cent. naph.	Per cent. kero.	Per cent. bot's.
Oklahoma:							
Beggs	32.7	120	Trace	15.00	8.60	13.20	63.20
Bixby	34.5	150	0.9	20.13	.....	20.35	59.52
Birdcreek	30.9	176	0.8	9.60	.....	15.00	75.00
Cement	36.1	110	....	15.00	7.50	17.50	60.00
Cushing	39.4	138	0.6	35.00	.....	15.00	50.00
Drumright	38.7	...	....	30.00	.....	20.00	50.00
Duncan	33.7	150	0.03	27.20	.....	15.00	57.80
Garber	47.1	117	0.80	55.00	.....	20.00	25.00
Hominy (Osage)	37.7	140	0.12	25.00	.....	20.00	55.00
Healdton	30.4	...	0.10	10.00	.....	23.00	67.00
Inola	36.2	...	2.00	27.50	.....	22.50	50.00
Jenks	32.8	220	0.30	15.00	.....	20.00	65.00
Madill	46.6	...	0.06	50.00	.....	20.00	30.00
Mervine	40.8	116	Trace	22.50	10.00	20.00	47.50
Mounds	34.7	...	....	20.00	.....	15.00	65.00
Quay	35.3	160	0.15	27.50	.....	15.00	57.50
Walters	29.8	...	....	25.00	.....	10.00	65.00
Youngstown	42.1	...	0.02	35.00	.....	20.00	45.00
Kansas:							
Augusta	41.1	136	0.10	30.00	.....	22.50	47.50
Chautauqua	35.5	126	0.60	27.50	.....	10.00	62.50
Greenwood Co.	34.5	...	0.34	15.00	.....	35.00	50.00
Iola	19.1	...	4.00	(Wax Dist. 50.00%—Bot's. 46.50%			
Louisiana:							
Pine Island	26.7	218	Trace	(Gas Oil 10.00%; Wax Dist. 87.00			
Texas:						Grav. 30.9; Flash	
Burkburnett	39.8	100	....	42.50	.....	7.50	50.00
Moran	34.7	170	0.46	10.00	11.00	17.50	61.50
Ranger	37.3	156	0.70	20.60	7.72	17.84	51.90
San Antonio	32.8	170	0.01	24.00	.....	12.50	63.50

Tests on products

Benzine			Naphtha			Kerosene			Bottoms		
Grav.	I.B.P.	Dry	Grav.	I.B.P.	Dry	Grav.	Flash	Fire	Grav.	Flash	Fire
Bé.	Fahr.	Fahr.	Bé.	Fahr.	Fahr.	Bé.	Fahr.	Fahr.	Bé.	Fahr.	Fahr.
59.3	132	440	49.5	222	...	41.7	130	156	24.4	290	335
57.3	134	448	....	...	...	42.5	128	148	26.3	250	284
51.3	166	458	....	...	...	43.9	RT	RT	28.6	255	235
58.1	156	504	49.8	258	510	43.7	144	172	28.0	275	340
58.2	132	460	....	...	...	42.2	132	166	25.7	290	330
57.9	138	457	....	...	...	43.3	124	150	27.2	270	330
59.2	140	490	....	...	...	42.6	140	168	21.7	265	305
60.9	127	451	....	...	...	42.8	135	164	27.7	310	370
58.8	136	444	....	...	...	43.7	110	130	27.6	260	300
57.9	118	442	....	...	...	42.5	RT	88	....	...	...
58.8	132	450	....	...	...	42.1	128	144	....	...	...
51.1	244	512	....	...	...	44.1	115	134	26.1	260	300
59.1	132	...	....	...	...	41.8	170	202	....	...	...
58.3	176	454	51.6	244	490	43.4	133	155	29.9	275	330
58.3	138	448	....	...	...	44.0	96	130	25.6	280	330
56.5	162	490	....	...	...	44.4	122	140	29.6	386	334
57.0	...	...	....	...	...	42.5	...	...	....	...	...
58.5	150	464	....	...	...	43.3	144	158	....	...	...
9.2	148	458	....	...	...	44.0	130	142	....	...	...
58.5	130	480	....	...	...	42.6	125	150	27.2	290	340
58.1	162	474	....	...	...	42.0	120	140	....	...	...

Loss 3.50%) (Wax Dist. Tests: Gr.30.3; Fl. 150; Fire 230; Visc. 51)

% Bot's 3.00%) (Gas Oil: Grav. 35.4; Flash 100; Fire 120) (Wax Dist.: 120; Fire 205; Cold 20; Vis. 42)

58.2	124	482	....	...	...	41.5	140	184	26.1	260	310
58.8	150	444	50.7	220	...	42.5	120	136	28.4	255	295
58.5	136	342	48.8	229	440	42.8	168	190	27.7	300	350
39.2	144	510	....	...	...	42.4	156	178	23.3	255	310

The product designated as bottoms in the table includes the gas oil and the product which contains the wax distillate, or lubricating stock, and which is sold as fuel oil by the skimming-plant operator.

Particularly interesting are the tests on Burkburnett, Ranger and Pine Island crudes and the analysis of crude from several wells in the San Antonio district of Texas, about which little information has been available hitherto.

The results shown in the table were obtained from laboratory distillation tests, made by running down 1000 c.c. of each crude. No losses are shown, but the loss runs from 1.5 to 3 per cent., with the average probably slightly above 1.5.

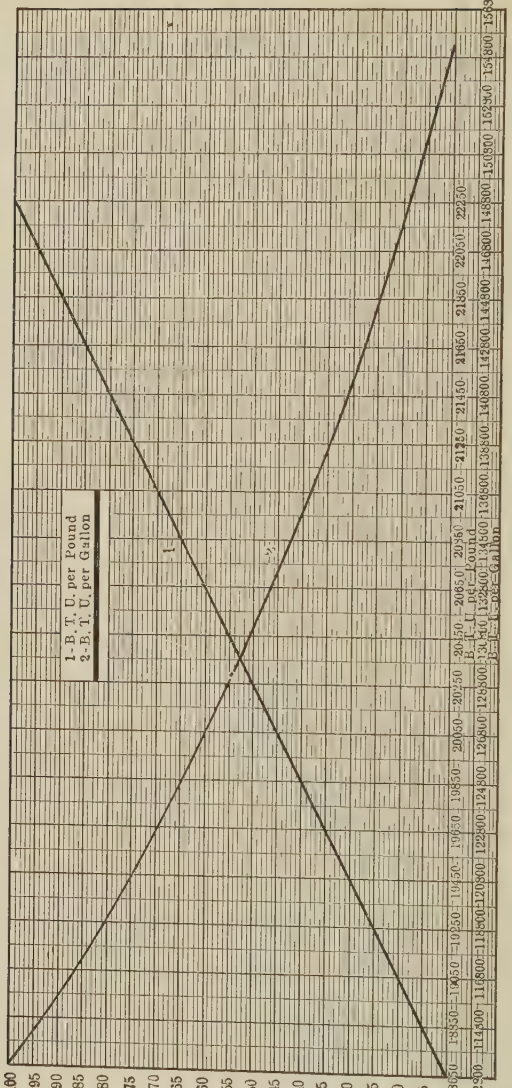


FIG. 116.—B.t.u. per pound and per gallon of oils of different Baumé gravities. The data are calculated from the formula B.T.U. = 18650 + 40(Bé.° − 10).

Physical Properties of the Chief Constituents of Gasoline

Fr. = French Physical Society Tables, 1910, 1911, 1912, and 1913.
H. = Hildebrand, J. Am. Chem. Soc., **39** (1917), 2126.
L. = Landolt-Börnstein, Physikalisch-Chemische Tabellen," **1912.**
Ol. = Olsen, Van Nostrand "Chemical Annual," 4th issue, **1918.**
S. = Bureau of Standards.
T. = Timmermans, Bull. Soc. Chem. Belg., **1911** and later years (several papers).

Name	Formula	Specific gravity at 20°C.	Freezing-point, °C.	Boiling-point, °C.	Heat of Combustion, cal./gram	Critical sol. temp., °C.	Rotatory power
Aromatics	C_nH_{2n-6}						
Benzene	C_6H_6	0.8786 Fr.10	5.6 L.	80.2 L.	10030 S.	Miscible	0.000550 S.
Toluene	C_7H_8	0.8657 Fr.10	− 94.5 T.	110.7 T.	10150 L.	do	0.000485 S.
Xylene-O	C_8H_{10}	0.8633 L.	− 27.1 L.	141.0 L.	10229 L.	do	
Xylene-M	C_8H_{10}	0.8642 L.	− 54.8 L.	139.0 T.	10228 L.	do	
Xylene-P	C_8H_{10}	0.8612 L.	+ 15.0 L.	137.5 L.	10229 L.	do	0.000446 S.
Naphthenes	C_nH_{2n}						
Cyclopentane	C_5H_{10}	0.751 L.	Below—80° L.	50.5 L.			
Methylcyclopentane	C_6H_{12}	.....	.....	.....	11237 L.		
Dimethylcyclopentane	C_7H_{14}	.....	.....	.....	11219 L.		
Cyclohexane	C_6H_{12}	0.7764 L.	+ 6.40 L.	80.9 L.	11217 L.	31.3 T.	0.000226 S.
Methylcyclohexane	C_7H_{14}	0.7695 Fr.11	.....	101.0 Fr.11	11151 L.	41.3 T.	
Cycloheptane	C_7H_{14}	(0.825 at 0°C.) L.	.....	(117° at 743 mm.)	10187 L.		
Methylcycloheptane	C_8H_{16}	.....	Very low L.				
Cyclooctane	C_8H_{16}						

Aliphatics (saturated)	C_nH_{2n+2}						
Pentane-normal	C_5H_{12}	(0.6454 at 0°) T.	−130.8 Fr.$_{11}$	36.3 Fr.$_{13}$	11650 S.		0.000211 S.
Isopentane	C_5H_{12}	0.6200 Fr.$_{10}$	−158.0 Fr.$_{10}$	27.9 Fr.$_{13}$			
Hexane-normal	C_6H_{14}	0.6694 Fr.$_{10}$	− 94.0 Fr.$_{11}$	69.0 Fr.$_{13}$	11501 L.	59.2 H	0.000247 S.
Isohexane	C_6H_{14}	0.6599 Fr.$_{10}$	.....	62.0 Fr.$_{13}$			
Heptane-normal	C_7H_{16}	0.733 Fr.$_{10}$	− 97.1 Fr.$_{11}$	98.4 Fr.$_{13}$	11374 L.		
Isoheptane	C_7H_{16}	(0.707 at 0°) Ol.	.....	90.3 Ol.			
Octane-normal	C_8H_{18}	(0.7185 at 0°) Fr.$_{13}$	− 56.5 Fr.$_{11}$	125.6 Fr.$_{13}$	1149 L.		
Isooctane	C_8H_{18}						
Olefines	C_nH_{2n}						
Amylene-normal	C_5H_{10}	0.6597 Fr.$_{12}$	.....	38.0 Fr.$_{12}$	11491 L.	21.8 T.	
Hexylene-normal	C_6H_{12}	0.6825 Ol.	− 98.5 Ol.	65.9 Fr.$_{13}$	11413 L.		
Heptylene-normal	C_7H_{14}	0.703 Ol.	.....	98.0 Ol.			
Octylene-normal	C_8H_{16}	0.7223 Ol.	.....	124.0 Ol.			

Government Gasoline Specifications

Aviation Gasoline. [U. S. Fuel Administration (1918).]

1. *Color.*—Water-white.

2. *Foreign Matter.*—Free from acid, undissolved water, and suspended matter.

3. *Doctor Test.*—Negative.

4. *Corrosion and Gumming Test.*—No gray or black deposit nor weighable amount of gum.

5. *Distillation* (Bureau of Mines Method).—In each case at least 96 per cent. shall be recovered and the distillation loss shall not exceed 2 per cent. when residue in the flask has been cooled and added to the distillate in the receiver.

(A) Export grade.
- 5 per cent. over between 50° and 65°C.
- 50 per cent. over at temperature not above 95°C.
- 90 per cent. over at temperature not above 125°C.
- 96 per cent. over at temperature not above 150°C.
- End-point not more than 15°C. above temperature corresponding to 96 per cent. of distillate.

(B) Fighting grade.
- 5 per cent. over from 60° to 70°C.
- 50 per cent. over at temperature not above 95°C.
- 90 per cent. over at temperature not above 113°C.
- 96 per cent. over at temperature not above 125°C.
- End-point not more than 15°C. above the temperature corresponding to 96 per cent. of distillate.

(C) Domestic grade.
- 5 per cent. over between 50° and 75°C.
- 50 per cent. over at temperature not above 105°C.
- 90 per cent. over at temperature not above 155°C.
- 96 per cent. over at temperature not above 175°C.

Motor Gasoline (1918).

1. *Quality.*—Must be high-grade product, refined, and free from water and all impurities.

2. *Distillation* (Bureau of Mines Method).
- Over-point not higher than 60°C. (140°F.).
- 20 per cent. over at or below 105°C. (221°F.).
- 45 per cent. over at or below 135°C. (275°F.).
- 90 per cent. over at or below 180°C. (356°F.).
- End- or dry-point not higher than 220°C. (428°F.).
- Not less than 95 per cent. shall be recovered from distillation.

Proposed Specifications for Motor Gasoline (1919).[1]

Distillation (Bureau of Mines Method).

Grade M.
- Over-point not above 60°C. (140°F.).
- 20 per cent. over at or below 105°C. (221°F.).

[1] See page 53.

50 per cent. over at or below 140°C. (284°F.).
90 per cent. over at or below 190°C. (374°F.).
Dry-point at or below 225°C. (437°F.).

Grade B.

Over-point not above 60°C. (140°F.).
20 per cent. over at or below 105°C. (221°F.).
50 per cent. over at or below 140°C. (284°F.).
90 per cent. over at or below 180°C. (356°F.).
Dry-point at or below 215°C. (419°F.).

Grade R.

Over-point not above 65°C. (149°F.).
20 per cent. over at or below 110°C. (230°F.).
50 per cent. over at or below 150°C. (302°F.).
90 per cent. over at or below 210°C. (410°F.).
Dry-point at or below 235°C. (455°F.).

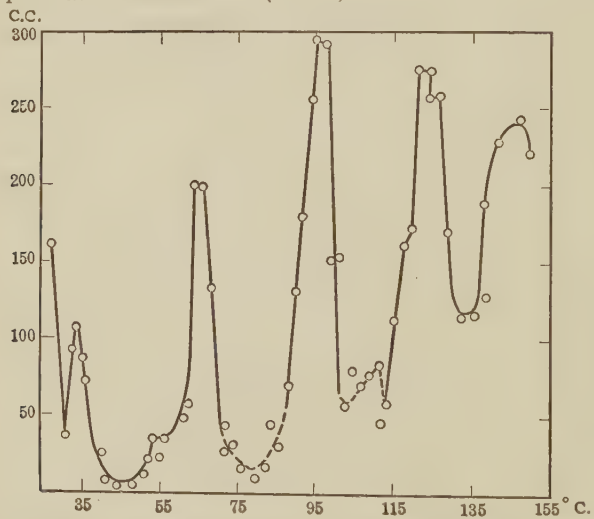


FIG. 117.—Fractional distillation curve of gasoline obtained by pressure distillation. (I W. Humphrey.)

Bureau of Explosives' Rules for the Shipment of Gasoline

CONDENSED INSTRUCTIONS FOR SHIPPERS OF GASOLINE, NAPHTHA, CASINGHEAD GASOLINE, AND LIQUEFIED PETROLEUM GAS

Statistics show these commodities, through their fire hazard, to be the most dangerous freight handled by the railroads of the United States. The loss of life and property has been caused principally by shipments in tank cars **not** prepared and loaded as required by the I. C. C. Regulations.

Hold one competent man at each loading point rigidly responsible for compliance with all rules and supply him and all other employees who have duties and responsibilities under them with a copy of the following condensed

instructions, which have been compiled for the purpose of assisting shippers to properly instruct their employees.

Marginal notes will assist in securing information quickly, while paragraph numbers will facilitate reference to the I. C. C. Regulations from which these condensed instructions were compiled.

Remember that when you neglect any one of the duties imposed on you by the regulations, you sign a false statement on your shipper's certificate; you violate the law framed to promote safety; you set a fire-trap that may kill or injure innocent men; and you injure your own business.

Consult I. C. C. Regulations.—These condensed instructions are only intended to assist shippers in educating their employees and in some instances it may be found necessary to consult the regulations for a full and complete understanding of the same.

Instruct Employees.—Paragraph 1703 of the I. C. C. Regulations states specifically that it is the duty of shippers to make the prescribed regulations effective and to thoroughly instruct their employees in relation thereto (1703).

Casinghead Products

Liquid Condensates, Description of.—Liquid condensates from natural gas or from casinghead gas of oil wells, made by the compression or absorption process, alone or blended with other petroleum products, require extra care in handling and are subject to certain special requirements according to the vapor pressure of the liquid. When the vapor pressure at 100°F. (90°F. Nov. 1st to March 1st) does not exceed 10 lb. per sq. in., they must be described as Gasoline, Casinghead Gasoline. When the vapor pressure exceeds 10 lb. per sq. in., they must be described as Liquefied Petroleum Gas (1824(k)).

Method of Making Vapor Pressure Tests.—Bureau of Explosives' circular M.C.L. No. 147 describes fully the method to be used in measuring the vapor tension of the material. In making this test it will be noted that the container may be vented momentarily at a temperature of 70°F., after which the tube must be placed in the water again for a period

Vapor pressure		Shipping description	Kind of shipping container required
Apr. 1 to Oct. 1	Oct. 1 to Apr. 1		
10 lb. pef sq. in. or less	10 lb. per sq. in. or less	Gasoline, Casinghead gasoline (use either)	Metal barrels or drums I. C. C.—5. Ordinary tank cars, Classes II and III tested to 60 lb. per sq. in. Insulated tank cars, Class IV tested to 75 lb. per sq. in.
Over 10 lb. and not exceeding 15 lb.	Over 10 lb. and not exceeding 20 lb.	Liquefied petroleum gas	Metal barrels or drums I. C. C.—5. Insulated tank cars, Class IV tested to 75 lb. per sq. in.
Over 15 lb. and not exceeding 25 lb.	Over 20 lb. and not exceeding 25 lb.	Liquefied petroleum gas	Metal barrels or drums I. C. C.—5.
Over 25 lb.	Over 25 lb.	Liquefied petroleum gas	Steel cylinders I. C. C.—26.

of ten minutes at a temperature of 100°F. From November 1st to March 1st, the temperature of the water in which the tube is placed for the last 10 minutes should be 90°F., but for the balance of the year the temperature must be maintained at 100°F. for the last 10 minutes the tube is in the water (1824(k)).[1]

The foregoing table shows the shipping description and kind of containers required for casinghead products, whether alone or blended with other materials such as naphtha, refined oil, crude oil, etc. It will be observed that four classes are provided, dependent upon the vapor pressure of the liquid, and for such liquids only those containers specified must be used (see par. 1824K). Where casinghead condensates are mixed with refinery products and the mixture passed through a refinery still, the finished product is not subject to these special rules for casinghead products, but is to be considered and shipped as straight gasoline, naphtha, etc., under the general rules for inflammable liquids (1824).

Refinery Products

Refinery Gasoline, Naphtha, Benzine, etc.—Refinery Gasoline, Naphtha, Benzine, etc. may be shipped in ordinary tank cars, 60 lb. test class complying with M.C.B. Specifications, or in metal barrels or drums I. C. C.—5, or in metal cans boxed I. C. C.—2, or in metal cans packed in fiberboard boxes I. C. C.—24, or in metal cans cushioned in wooden barrels I. C. C.—11, or in wooden barrels or kegs I. C. C.—9, or in metal jacketed cans, I. C. C.—23 (1824 (a) to (f) incl.).

Tests of Ordinary Tank Cars and Safety Valves.—The Master Car Builders' Specifications require that ordinary tank cars, 60 lb. test class, must have their shells retested within ten years after being placed in service, and thereafter every five years, and safety valves must be set at 25 lb. and retested not less than every two years (M.C.B. Spec. Sections 23 & 24).

Tests of Insulated Tank Cars and Safety Valves.—Insulated tank cars must be tested with cold water pressure at 75 lb. per sq. in., and safety valves set to operate at 25 lb. per sq. in. Shells must be retested every five years and safety valves every six months (M.C.B. Spec. Sections 23 & 24).

Stenciling Tank Cars.—Both ordinary and insulated tank cars must be properly stenciled to show date and place of test and by whom made (1822D & M.C.B. Spec. Fig. 6).

Shipper's Responsibility.—When tank cars are used, shipper must know that they comply with Master Car Builders' Specifications. A tank car not tested (shell or safety valve) within the limits prescribed by the Master Car Builders' Rules must not be used (1822, 1824 and M.C.B. Rules).

Specifications.—Copy of Master Car Builders' Specifications may be obtained from E. R. Hawthorne, Secretary, Section III—Mechanical, American Railroad Association, Manhattan Building, Chicago, Ill.

Tank Cars that Must not be Used.—A tank that does not meet the prescribed tests must be withdrawn from transportation service (M.C.B. Rules).

Dome Covers Must be "Fool-proof."—Dome covers that cannot be removed while pressure exists in tank, or that will warn any one attempting to remove the dome cover of the fact that pressure exists in the tank, must be used (1824 (k)).

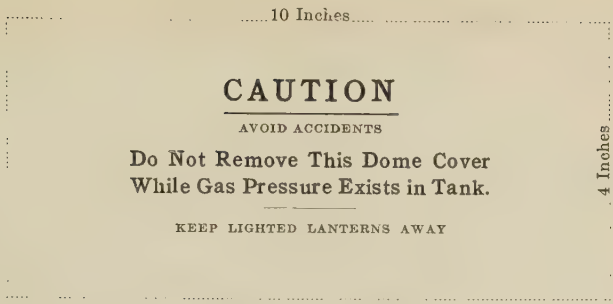
Examination of Tank Cars and Precautions Necessary When Loading.—Examine tanks and their fittings carefully to see that they are in proper condition before loading. **Load with valve cap off.** After loading outlet valves must not permit more than a dropping of the liquid with the valve cap off, otherwise valve must be reground and repaired. If dropping appears at seams or rivets such leak must be properly repaired by calking. Dome covers must be tight against vapor pressure and, when necessary, must be provided with suitable gaskets and must be forced tightly into place before cars are tendered to the carrier. When practicable, it is recommended that valve caps be allowed to remain off until carriers' car inspector has passed on the loaded car (1822 (e)).

Do Not Forward Leaking Tank Cars.—Under no circumstances must a tank car be allowed to go forward that leaks or that has any defect that would make leakage during transit probable (1822 (d)).

Inflammable Placards.—The shipper must attach four regulation "INFLAMMABLE" placards, one on each side and one on each end of the tank car (1913).

Special Dome Placards for Liquid Condensates.—For Casinghead products the shipper must also attach securely and conspicuously to the dome and dome cover three white dome placards measuring 4 × 10 in., bearing the following wording:

[1] Paragraph references are figures in parentheses.



One placard must be attached to each side of the dome and one placard must be attached to the dome cover (1824 (k)).

Minimum Outage Requirements.—Not less than 2 per cent. of the total capacity of all containers, including tank cars, must be left vacant to allow for the expansion of contents during transit. If the dome of tank cars does not provide this 2 per cent., sufficient vacant space must be left in the shell of the tank to make up the difference (1825).

An outage of 2 per cent. is frequently insufficient for light petroleum products, owing to the fact that they are generally cold when loaded, they expand more than heavier petroleum products when the temperature increases and this rate of expansion varies with the specific gravity of the material. It is recommended that when tank cars are loaded with liquid condensates properly described as Gasoline, Casinghead Gasoline, Casinghead products or blends (see Par. 1824 (k)), the outage in tank shall not be less than the following (1825).

OUTAGE TABLE FOR CASINGHEAD PRODUCTS

Temperature of product when loaded	Minimum outage required when gravity is			
	50–60°Bé., per cent.	60–70°Bé., per cent.	70–80°Bé., per cent.	
0–60°F	3.2	3.5	4.1	
61–70°F	2.5	2.8	3.3	
71–80°F	2.0	2.1	2.4	
81–100°F	2.0	2.0	2.0	(1825)

Adjustment of Outage in Loaded Tank Cars.—Before closing dome of the loaded tank car, measure temperature of liquid and, when necessary, remove liquid to adjust outage. For example: shell capacity 10,000 gal.; dome capacity 2 per cent. when loaded shell full; gravity 75°Bé.; temperature 40°F. From table required outage is 4.1 per cent. Since 10,000 gal. equal 98 per cent. of combined shell and dome capacity, 1 per cent. equals 102 gal. 2 per cent. or dome capacity 204 gal and 4.1 per cent. or required outage 418 gal. Number of gallons to be removed from car for adjustment of outage is therefore 214 gallons.

Leaks at Bungs Must be Prevented.—Every precaution must be taken to prevent leaks at bungs. Tight-fitting bungs and proper gaskets must be used, and it is recommended that the gasket and flange of the bung be coated with gum shellac dissolved in alcohol to about the consistency of molasses and then the bung screwed tightly into place with a long handled wrench (not less than 18 in. long). The barrel should then be allowed to stand on end with bung up until the shellac dries, after which it should be placed on its side with bung and then filling plug down to test for leakage, allowing ample time for the tests. The term bung, as used above, includes filling or vent plugs, and if bung is on side of barrel, the same precaution should be taken to prevent leakage during transit. Under no circumstances allow a leaking package to leave your establishment (1801C & Spec. No. 5).

SPECIAL LABEL RECOMMENDED FOR USE ON BARRELS OR DRUMS CONTAINING LIQUEFIED PETROLEUM GAS

CAUTION

Unscrew This Bung SLOWLY.

Do not unscrew it entirely until all interior pressure is removed by escape of vapor through loosened threads. Remove bung in open air, keeping all open lights and fires away.

CLOSED ELECTRIC LIGHTS ARE SAFE

Manufacturers' Circular Letter No. 265.

Marking Required to Show Compliance with I. C. C. Specifications.—All outside containers must be marked to show compliance with the specifications as shown in the specifications that apply. When metal barrels or drums are used they must comply with I. C. C. Specification No. 5 and each barrel or drum must be permanently marked to show compliance with the specification thus:

"Complies with I. C. C. Specification No. 5" or the marking "I. C. C.—5" and with the name of manufacturer and date of manufacture. The above system of marking applies to wooden boxes, and strawboard or fiberboard boxes, which are marked "I. C. C.—2" and "I. C. C.—24," respectively, and to metal jacketed cans, I. C. C.—23. Wooden barrels are marked "I. C. C.—9" or "I. C. C.—11," according to the specification under which they are manufactured, except that name of manufacturer and date of manufacture are not required on containers other than I. C. C. No. 5 (1822)

Specifications.—Specifications for each of the above containers will be found in the "Red Book" (B. E. Pamphlet No. 9) and also in B. E. Pamphlet No. 11.

Marking Required to Show Name of Contents.—All barrels, drums, boxed cans or other packages must be marked or stenciled to show nature of contents, thus: "Gasoline," "Benzine," "Naphtha," "Casinghead Gasoline," or "Liquefied Petroleum Gas," etc. (1712)

This Side Up.—Interior packages of 1 quart or more must be packed with their filling holes up and the outside package must be plainly marked **"This Side Up"** (1826).

Name and Address of Consignee. Labels and Placards Required.—In less than carload shipments each barrel, drum, boxed can or other package must be marked with name and address of consignee and each must bear a red label. Red labels may be omitted on carload shipments to be unloaded by the consignee, but proper "Inflammable" placards must be secured from the carrier and applied to the car by the shipper (1712) (1864 (b)).

Tank Car Marking.—Loaded tank cars must bear thereon a card showing proper name of contents (1712).

Shipping Order Requirements.—Shipping orders must describe shipments under proper classification or tariff name and for less than carload shipments must show color of label applied, opposite entry for which intended, while for carload shipments the kind of placard applied to the car should be shown thus: (1712 & 1867).

Gasoline	Red Label
Benzine	Red Label
Naphtha	Red Label
Casinghead Gasoline	Red Label
Liquefied Petroleum Gas	Red Label
or for carloads	
Gasoline	Inflammable Placard
Benzine	Inflammable Placard
Naphtha	Inflammable Placard
Casinghead Gasoline	Inflammable Placard
Liquefied Petroleum Gas	Inflammable Placard

Shipper's Certificate.—Shipping orders must also show proper shipper's certificate in lower left-hand corner over shipper's signature (1867C).

Below will be found sample of a properly prepared shipping order, bearing proper certificate over shipper's written signature.

EXPRESS SHIPMENTS

Express Shipments Limited.—Express shipments of refinery and casinghead product, must not be made in outside packages containing more than one gallon and must be packeds marked and labeled in accordance with the following condensed instructions (List 34, Col. 4, General Notice F, General Rule I).

Description of Inside Containers and Capacity Limits.—Inside containers must consist of well-stoppered glass or earthenware vessels, not exceeding one quart capacity each, or tightly closed metal cans not exceeding one gallon capacity each (List 34, Col. 4, 39 (b), (c)).

Outside Containers Required for Inside Containers.—Inside containers not exceeding one pint capacity each must be packed or cushioned in outside containers complying with I. C. C. Specifications No. 2, No. 11, or No. 24 (List 34, Col. 4, 39 (d)).

Inside containers not exceeding one quart capacity each must be packed or cushioned in outside containers complying with I C. C. Specifications No. 2, or No. 11 (List 34, Col. 4, 39 (b), (c)).

Metal cans not exceeding one gallon must be packed or cushioned in outside containers complying with I. C. C. Specifications No. 2 or No. 11 (List 34, Col. 4, 37, 39 (b)).

Other Authorized Containers.—Shipments may be made in metal jacketed cans not exceeding one gallon capacity complying with I. C. C. Specification No. 23 (List 34, Col. 4, 39 (e), (f)).

Minimum Outage Requirement.—Not less than 2 per cent. of the total capacity of all containers must be left vacant to allow for the expansion of contents during transit (38 (a)).

Casinghead Gasoline. Liquefied Petroleum Gas.—Casinghead Gasoline (Liquefied Petroleum Gas,blended or unblended), when its vapor pressure exceeds 10 lb. per sq. in., must not be shipped by express, except in steel cylinders conforming to paragraphs 57, 58 and 59 (39 (h)).

Markings and Label Required.—Each outside package must also be marked with proper name of contents, name and address of consignee, specification marking as shown in the different specifications, and a red label as prescribed for express shipments must be applied (1, 36 (a), 60, 61).

This Side Up.—Interior packages containing one pint or more must be packed with their filling holes up and the top of the outside package must be plainly marked "This Side Up" (36 (d)).

No Exemption from Label.—There is no exemption from labels when shipped by express, on account of small quantities or for any other reason (60 (a)).

EXPRESS LABELS

Extra Certificate When Freight Labels are Used.—Attention is called to the fact that express labels differ from freight labels inasmuch as the express labels bear shipper's certificate, whereas freight labels do not. Shippers desiring to take advantage of paragraph 62 of the express regulations may use the label prescribed for freight shipments, provided extra certificate is stamped or pasted on package alongside of label. Certificates on express labels must be signed by shipper as must also the extra certificate when used (61–62).

NOTICE

The Interstate Commerce Commission has revised, effective December 15, 1919, paragraph 1824 (k) of the regulations. The revised paragraph reads as follows:

1824 (k). Liquid condensates from natural gas or from casinghead gas of oil wells, made either by the compression or absorption process, alone or blended with other petroleum

products, must be described as Liquefied Petroleum Gas when the vapor pressure[1] at 100°F. (90°F. November 1 to March 1) exceeds 10 lb. per square inch.

When the liquid condensate alone or blended with other petroleum products has a vapor pressure not exceeding 10 lb. per sq. in., it must be described and shipped as Gasoline, or Casinghead Gasoline.

Liquefied petroleum gas of vapor pressure exceeding 10 lb. per sq. in. and not exceeding 15 lb. per sq. in., from April 1 to October 1, and 20 lb. per sq. in., from October 1 to April 1, must be shipped in metal drums or barrels which comply with Shipping Container Specification No. 5; or in special insulated tank cars approved for this service by the Master Car Builders' Association.

Liquefied petroleum gas of vapor pressure exceeding 15 or 20 lb. per sq. in. as provided herein, and not exceeding 25 lb. per sq. in., must be shipped only in metal drums or barrels which comply with Shipping Container Specification No. 5.

Liquefied petroleum gas of vapor pressure exceeding 25 lb. per sq. in. must be shipped in cylinders as prescribed for compressed gases (see pars. 1861 to 1863, inclusive).

When the liquid condensate, alone or blended with other petroleum products, has a vapor pressure not exceeding 10 lb. per sq. in., it must be described as Gasoline or Casinghead Gasoline and must be shipped in metal drums or barrels complying with Specification No. 5; or in ordinary tank cars, 60 lb. test class equipped with mechanical arrangement for closing of dome covers as specified in Master Car Builders' specifications for tank cars.

Every tank car containing liquid condensates, either blended or unblended, including liquefied petroleum gas, as defined herein, must have safety valves set to operate at 25 lb. per sq. in. with a tolerance of 3 lb. above or below, and the mechanical arrangements for closing the dome covers of such cars must either be such as to make it practically impossible to remove the dome cover while the interior of the car is subjected to pressure; or suitable vents that will be opened automatically by starting the operation of removing the dome cover must be provided.

The shipper must attach securely and conspicuously to the dome and dome cover three special white dome placards measuring 4 × 10 in., bearing the following wording:

10 Inches

CAUTION

AVOID ACCIDENTS

Do Not Remove This Dome Cover While Gas Pressure Exists in Tank.

KEEP LIGHTED LANTERNS AWAY

4 Inches

One placard must be attached to each side of the dome and one placard must be attached to the dome cover. The presence of these special dome placards must be noted on the shipping order by the shipper and by the carrier on the billing accompanying the car. Placards must conform to samples furnished by the Chief Inspector of the Bureau of Explosives.

Note to paragraph 1825 (a) of the regulations has also been amended, effective December 15, 1919. This paragraph now reads as follows:

1825 (a) Packages containing inflammable liquids must not be entirely filled. Sufficient interior space must be left vacant to prevent leakage or distortion of containers due to increase of temperature during transit. In all such packages this vacant space must not be less than 2 per cent. of the total capacity of the container. In tank cars the vacant

[1] In measuring the vapor pressure the container may be vented momentarily at a temperature of 70°F.

SAMPLE SHIPPING ORDER

NORTH & SOUTH RAILROAD COMPANY

THIS SHIPPING ORDER must be legibly filled in, in Ink, in Indelible Pencil, or in Carbon, and retained by the Agent.

SHIPPERS NO. _____

AGENTS NO. _____

RECEIVE, subject to the classifications and tariffs in effect on the date of issue of this Shipping Order.

at New York City, N. Y. July 25, 191 9

from Richard Roe & Co. the property described below, in apparent good order, except as noted (contents and condition of contents of packages unknown), marked, consigned and destined as indicated below, which said Company agrees to carry to its usual place of delivery at said destination, if on its road, otherwise to deliver to another carrier on the route to said destination. It is mutually agreed, as to each carrier of all or any of said property over all or any portion of said route to destination, and as to each party at any time interested in all or any of said property that every service to be performed hereunder shall be subject to all the conditions, whether printed or written, herein contained (including conditions on back hereof) and which are agreed to by the shipper and accepted for himself and his assigns.

The Rate of Freight from N. Y.

to Chicago, Ill. *is in Cents per 100 Lbs.*

If ... Times 1st	If 1st Class	If 2d Class	If Rule 25	If 3d Class	If Rule 28	If Rule 29	If 4th Class	If 5th Class	If 6th Class	If Special per ...	If Special per ...

(Mail Address—Not for purposes of Delivery.)

Consigned to John Doe & Co.

Destination, Chicago State of Ill. County of Cook.

Route, N & R RR and S & N RR Car Initial N & S Car No. 4113

NO. PACKAGES	DESCRIPTION OF ARTICLES AND SPECIAL MARKS	WEIGHT Subject to Correction	CLASS OR RATE	CHECK COLUMN
3	Boxes Gasoline, in cans (Red Label)	270		
1	Case Gasoline, in Cans (Red Label)	30		
1	Bbl. Gasoline, in cans (Red Label)	320		
1	Wooden Bbl. Gasoline (Red Label)	410		
2	Metal Drums Gasoline (Red Label)	1050		
4	Metal Bbls. Casinghead Gasoline (Red Label)	2100		
		525		
1	Drum Liquefied Petroleum Gas (Red Label)	515		
1	Cylinder Liquefied Petroleum Gas (Red Label)	150		
	This is to certify that the above articles are properly described by name and are packed and marked and are in proper condition for transportation, according to the regulations prescribed by the Interstate Commerce Commission.			

If charges are to be prepaid, write or stamp here, "To be Prepaid."

Received $ ..... to apply in prepayment of the charges on the property described hereon.

Agent or Cashier

Per .....

(The signature here acknowledges only the amount prepaid.)

Charges Advanced:

$ _____

RICHARD ROE & CO. Shipper

Per John Smith

☞ Agent must detach and retain this Shipping Order and must sign the Original Bill of Lading.

SAMPLE SHIPPING ORDER

NORTH & SOUTH RAILROAD COMPANY

SHIPPERS NO. .....

THIS SHIPPING ORDER must be legibly filled in, in Ink, in Indelible Pencil, or in Carbon, and retained by the Agent.

AGENTS NO. .....

RECEIVE, subject to the classifications and tariffs in effect on the date of issue of this Shipping Order

at New York City, N. Y. July 25, 191 9

from Richard Roe & Co. the property described below, in apparent good order, except as noted (contents and condition of contents of packages unknown), marked, consigned and destined as indicated below, which said Company agrees to carry to its usual place of delivery at said destination, if on its road, otherwise to deliver to another carrier on the route to said destination. It is mutually agreed, as to each carrier of all or any of said property over all or any portion of said route to destination, and as to each party at any time interested in all or any of said property that every service to be performed hereunder shall be subject to all the conditions, whether printed or written, herein contained (including conditions on back hereof) and which are agreed to by the shipper and accepted for himself and his assigns.

The Rate of Freight from N. Y.

to Chicago, Ill. *is in Cents per 100 Lbs.*

If . . Tissas 1st	If 1st Class	If 2d Class	If Rule 25	If 3d Class	If Rule 26	If Rule 28	If 4th Class	If 5th Class	If 6th Class	If Special per.....	If Special per.....

(Mail Address—Not for purposes of Delivery.)

Consigned to John Doe & Co.

Destination, Chicago State of Ill. County of Cook.

Route, N & S RR and S & N RR Car Initial NSX 1123 / NSX 4113 / NSX 4115 Car No.

NO. PACKAGES	DESCRIPTION OF ARTICLES AND SPECIAL MARKS	WEIGHT Subject to Correction	CLASS OR RATE	CHECK COLUMN
1 Tank Car	8055 gals. Gasoline (Inflammable Placards) Outage 2%	53163		
1 Tank Car	7989 gals. Casinghead Gasoline (Inflammable & Dome Placards) Gravity 63 B Temperature 65 F Shell Capacity 8014 gals. Dome " 205 " Outage 2.8%	52727		
1 Tank Car	8011 gals. Liquefied Petroleum Gas (Inflammable & Dome Placards) Gravity 81.8 B Temperature 48 F Shell Capacity 8092 gals. Dome " 262 " Outage 4.1%	52457		
	This is to certify that the above articles are properly described by name and are packed and marked and are in proper condition for transportation, according to the regulations prescribed by the Interstate Commerce Commission.			

If charges are to be prepaid, write or stamp here, "To be Prepaid."

Received $..... to apply in prepayment of the charges on the property described hereon.

Agent or Cashier

Per .....

(The signature here acknowledges only the amount prepaid.)

Charges Advanced:

$.....

RICHARD ROE & CO. Shipper

Per John Smith

☞ Agent must detach and retain this Shipping Order and must sign the Original Bill of Lading.

space must not be less than 2 per cent.[1] of the total capacity of the tank, *i.e.*, the shell and dome capacity, combined. If the dome of tank cars does not provide this 2 per cent., sufficient vacant space must be left in the shell of the tank to make up the difference.

You will note from the foregoing that the descriptive term "Casinghead Naphtha" has been eliminated from both paragraph 1824 (k) and the note to paragraph 1825 (a).

B. W. DUNN,
Chief Inspector.

Standard Tests for Paint Thinners Other Than Turpentine, Adopted by the American Society for Testing Materials

SPECIFIC GRAVITY

The specific gravity may be determined by a hydrometer, Westphal balance, or pycnometer, provided these instruments are verified. The observations shall be taken with the sample at 15.56°C. (60°F.) and compared with water at the same temperature.

DISTILLATION

Flask.—The flask used shall be the standard Engler flask as described in the various standard works upon petroleum, such as Redwood, Holde, etc.

"Engler employs a globular flask 6.5 cm. in diameter, with a cylindrical neck 1.6 cm. in internal diameter and 15 cm. in length, from the side of which a vapor tube 10 cm. in length extends at an angle of 75 deg. downwards to the condenser. The junction of the vapor tube with the neck of the flask should be 9 cm. above the surface of the oil when the flask contains its charge of 100 c.c. of oil. The observance of the prescribed dimensions is considered essential to the attainment of uniformity of results."[2]

The flask shall be supported in a ring of asbestos having an opening 1¼ in. in diameter in its center.

The flask, burner, etc., shall be surrounded by a shield.

Condenser.—The condenser shall consist of a tube of thin brass of ½ in. internal diameter, 22 in. in length, set at an angle of 75 deg. with the flask,

[1] An outage of 2 per cent. is frequently insufficient for light petroleum products, owing to the fact that they expand more than heavier petroleum products when the temperature increases, and this rate of expansion varies with the specific gravity of the material. It is recommended that when tank cars are loaded with gasoline or casinghead gasoline (see Par. 1824 (k)) the outage in tank shall not be less than the following:

Temperature of product when loaded	Minimum outage required when gravity is		
	50–60°Bé., per cent.	60–70°Bé., per cent.	70–80°Bé., per cent.
0–60°F	3.2	3.5	4.1
61–70°F	2.5	2.8	3.3
71–80°F	2.0	2.1	2.4
81–100°F	2.0	2.0	2.0

[2] Redwood, 3d edition, Vol. 2, p. 205 (1913).

and surrounded by a water jacket of the trough type. The lower end of the condenser shall be cut off at an acute angle and shall be curved down for a length of 3 in., so as to project at least ½ in. into the 100-c.c. cylinder used as a receiver.

A cover (pasteboard) should be placed over the top of the cylinder and surrounding the condenser tube.

Thermometer.—The thermometer used shall conform to the following specifications.

The thermometer shall be graduated from 0 to 400°C. in intervals of 1°C. There shall be a small reservoir above the 400° mark. The thermometer shall be finished at the top with a small glass ring. The stem shall be made of enamel-backed thermometer tubing, but not of Jena 16^{III} glass. The bulb shall be made of Jena 16^{III}, Corning normal, or Jena or Corning borosilicate glass.

Every fifth graduation shall be longer than the intermediate ones, and the marks shall be numbered at every interval of 10°. The graduation marks shall be clear cut and fine, and the numbering clear cut and distinct. The thermometer shall be filled above the mercury with an inert gas, which will not act chemically on or contaminate the mercury. The pressure of the gas shall be sufficient to prevent separation of the mercury column at all temperatures of the scale, but the upper reservoir shall be large enough so that the pressure will not become excessive at the highest temperature.

The thermometer shall be thoroughly annealed before the final filling. It shall be pointed for use at full immersion.

Each thermometer shall be provided with a suitable case. A serial number for identification and the word "Distillation" shall be engraved on the stem.

All material and workmanship shall be of the best grade.

The maximum error from 0 to 200°C. shall not exceed 0.5°C.; from 200 to 300°C., shall not exceed 1°C.; and from 300 to 400°C., shall not exceed 2°C.

The thermometer shall conform to the following dimensions:

Total length, max., mm	385
Diameter of stem, mm	5.5–7.0
Diameter of bulb, mm	5.5–7.0
Diameter of capillary, min., mm	0.1
Length of bulb, mm	11–15
Distance, 0° to bottom of bulb, mm	25–35
Distance, 0 to 400° mark, mm	280–300

The thermometer shall be inserted through a tight fitting cork in the neck of the flask, so that the top of the thermometer bulb will be on a level with the bottom of the side outlet in the neck of the flask and in the center of the neck.

Method of Distillation.—The flask, connected with the condenser, shall be filled with 100 c.c. of the thinner at 60°F., which shall be measured in the 100-c.c. receiving cylinder. The same cylinder may be used without drying as the receiving vessel for the distillate. The flask shall be heated directly by a suitable burner.

The distillation shall proceed at the rate of not less than 4 nor more than 5 c.c. per minute into the receiving cylinder. The temperature at which the first drop leaves the lower end of the condenser shall be considered the initial boiling-point.

Readings of the quantity in the receiver shall be taken when the next 10° point is reached, and for every even 10° thereafter. For example, if initial boiling-point occurs at 144°C., then the first reading of the quantity in the receiver shall be made at 150°C., and thereafter at 160°, 170°, etc.

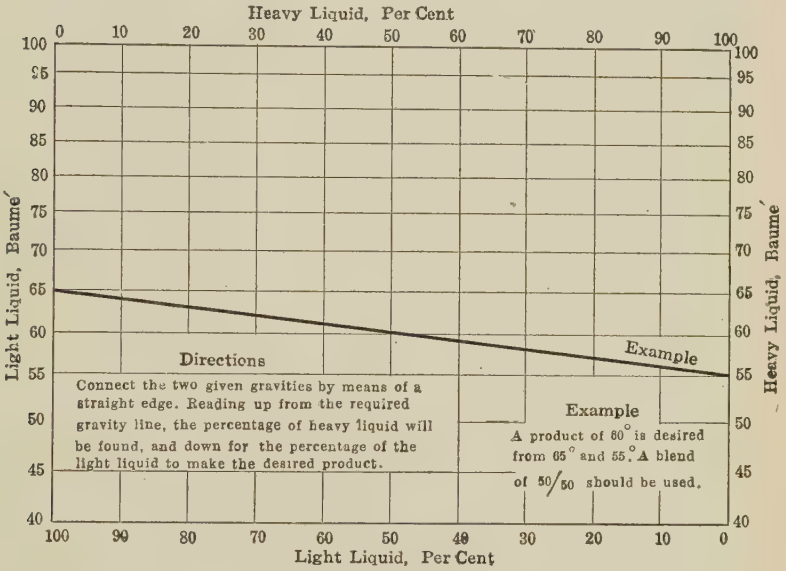

Heavy Liquid, Per Cent
0 10 20 30 40 50 60 70 80 90 100
100 95 90 85 80 75 70 65 60 55 50 45 40
Light Liquid, Baumé
Heavy Liquid, Baumé
Directions
Connect the two given gravities by means of a straight edge. Reading up from the required gravity line, the percentage of heavy liquid will be found, and down for the percentage of the light liquid to make the desired product.
Example
Example
A product of 60° is desired from 65° and 55°. A blend of 50/50 should be used.
100 90 80 70 60 50 40 30 20 10 0
Light Liquid, Per Cent

FIG. 118.—Blending chart for gasoline. Cosden & Company Laboratory, 1919).

The distillation shall be continued until the point is reached where the last drop is vaporized, when a puff of white vapor usually appears in the bottom of the flask. The temperature at this point shall be considered the end- or dry-point of distillation.

The total yield of distillate should not be less than 97 per cent.

DESCRIPTIVE NOTE TO FIG. 119.

On the line *A-A* locate the point representing the Baumé gravity of the casinghead. On the line *B-B* similarly locate the point representing the gravity of the blending naphtha. By means of a cord or straight-edge connect these points with a straight line. From the point where this line crosses the horizontal line representing the gravity desired in the finished product, follow the vertical line to the base, where the gallons of naphtha to be used for each gallon of casinghead are indicated.

If it is desired to know the gallons of casinghead per gallon of naphtha, use the line *A-A* for the naphtha gravity and *B-B* for the casinghead gravity. The base line will then indicate gallons of casinghead per gallon of naphtha.

This chart allows for the fact that the value of the Baumé degree is not constant throughout its scale.

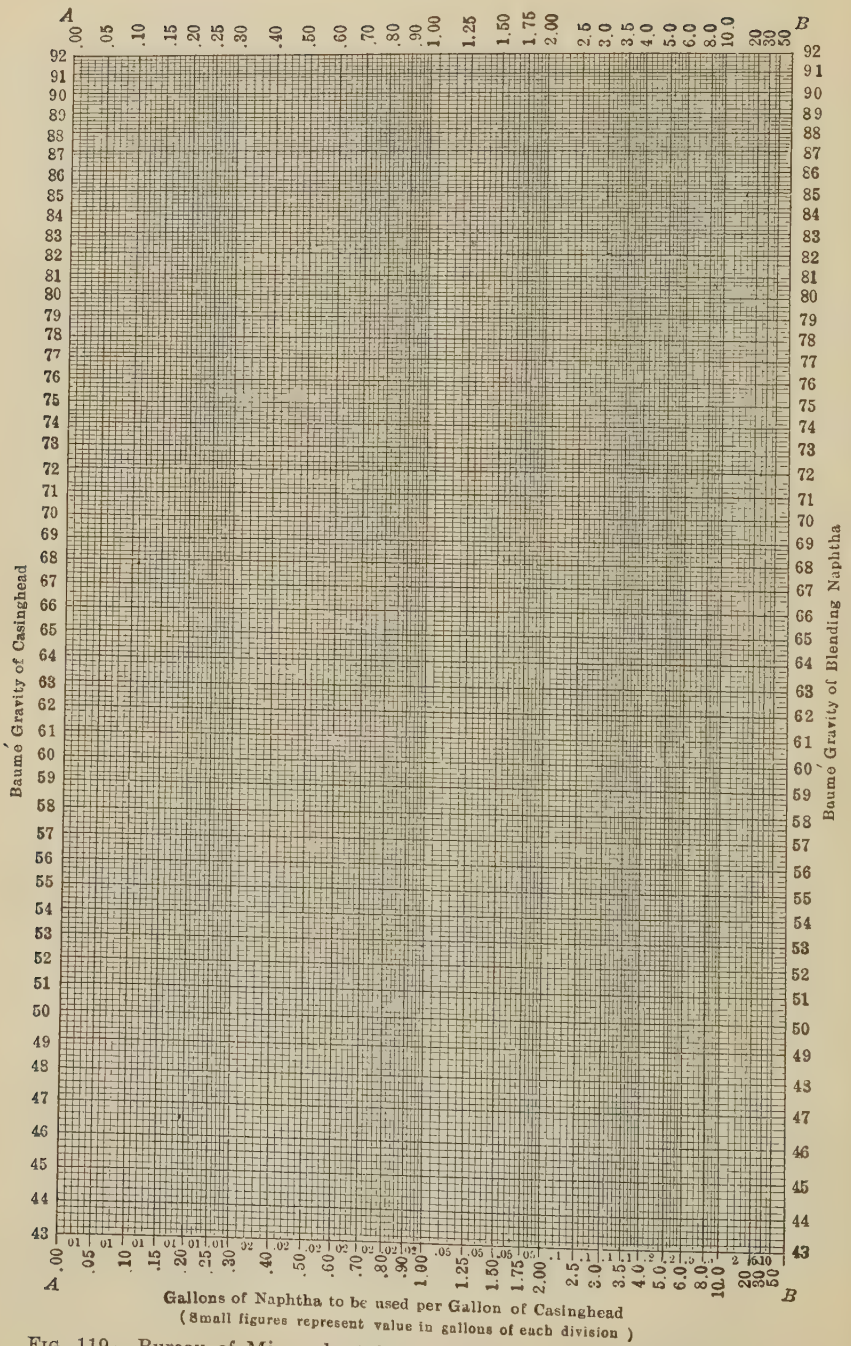
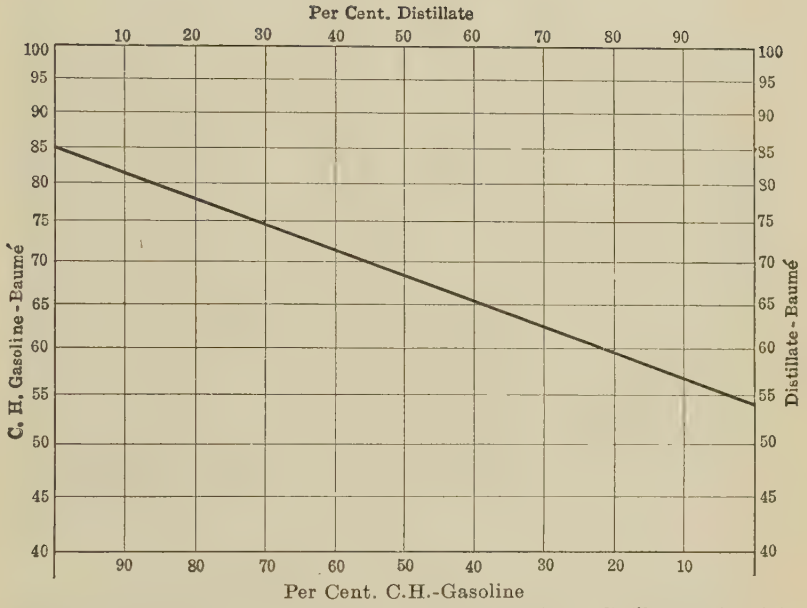

A
B
Baumé Gravity of Casinghead
Baumé Gravity of Blending Naphtha
Gallons of Naphtha to be used per Gallon of Casinghead
(Small figures represent value in gallons of each division)

FIG. 119.—Bureau of Mines chart for blending casinghead and naphtha to a desired gravity (computed by Clarence Netzen).

(See descriptive note on page 345.)


Per Cent. Distillate
10 20 30 40 50 60 70 80 90
100 95 90 85 80 75 70 65 60 55 50 45 40
C. H. Gasoline - Baumé
Distillate - Baumé
90 80 70 60 50 40 30 20 10
Per Cent. C.H.-Gasoline

Directions: Connect distillate gravities on right, with C.H. gasoline, on left, with a straight edge. Where straight edge crosses required blend gravity, "horizontal line," read on vertical line: upper margin, % distillate; lower, % C.H. gasoline.

Example: 54° Bé. distillate; and 58° Bé. C.H. gasoline. Make 70° Bé. blend, by using 45% distillate and 55% C.H. gasoline.

FIG. 120.—Blending chart for gasoline.

Correction of Flash-Point for Normal Barometric Pressures

[Reading of barometer, mm.]

Flash-point, °C.[1]

760	650	655	660	665	670	675	680	685	690	695	700	705	710	715
19.0	15.5	15.6	15.7	15.8	15.9	16.1	16.2	16.4	16.6	16.7	16.9	17.1	17.3	17.4
19.5	16.0	16.1	16.2	16.3	16.4	16.6	16.7	16.9	17.1	17.2	17.4	17.6	17.8	17.9
20.0	16.5	16.6	16.7	16.8	16.9	17.1	17.2	17.4	17.6	17.7	17.9	18.1	18.3	18.4
20.5	17.0	17.1	17.2	17.3	17.4	17.6	17.7	17.9	18.1	18.2	18.4	18.6	18.8	18.9
21.0	17.5	17.6	17.7	17.8	17.9	18.1	18.2	18.4	18.6	18.7	18.9	19.1	19.3	19.4
21.5	18.0	18.1	18.2	18.3	18.4	18.6	18.7	18.9	19.1	19.2	19.4	19.6	19.8	19.9
22.0	18.5	18.6	18.7	18.8	18.9	19.1	19.2	19.4	19.6	19.7	19.9	20.1	20.3	20.4
22.5	19.0	19.1	19.2	19.3	19.4	19.6	19.7	19.9	20.1	20.2	20.4	20.6	20.8	20.9
23.0	19.5	19.6	19.7	19.8	19.9	20.1	20.2	20.4	20.6	20.7	20.9	21.1	21.3	21.4
23.5	20.0	20.1	20.2	20.3	20.4	20.6	20.7	20.9	21.1	21.2	21.4	21.6	21.8	21.9
24.0	20.5	20.6	20.7	20.8	20.9	21.1	21.2	21.4	21.6	21.7	21.9	22.1	22.3	22.4
24.5	21.0	21.1	21.2	21.3	21.4	21.6	21.7	21.9	22.1	22.2	22.4	22.6	22.8	22.9
25.0	21.5	21.6	21.7	21.8	21.9	22.1	22.2	22.4	22.6	22.7	22.9	23.1	23.3	23.4
25.5	22.0	22.1	22.2	22.3	22.4	22.6	22.7	22.9	23.1	23.2	23.4	23.6	23.8	23.9
26.0	22.5	22.6	22.7	22.8	22.9	23.1	23.2	23.4	23.6	23.7	23.9	24.1	24.3	24.4
26.5	23.0	23.1	23.2	23.3	23.4	23.6	23.7	23.9	24.1	24.2	24.4	24.6	24.8	24.9
27.0	23.5	23.6	23.7	23.8	23.9	24.1	24.2	24.4	24.6	24.7	24.9	25.1	25.3	25.4
27.5	24.0	24.1	24.2	24.3	24.4	24.6	24.7	24.9	25.1	25.2	25.4	25.6	25.8	25.9
28.0	24.5	24.6	24.7	24.8	24.9	25.1	25.2	25.4	25.6	25.7	25.9	26.1	26.3	26.4
28.5	25.0	25.1	25.2	25.3	25.4	25.6	25.7	25.9	26.1	26.2	26.4	26.6	26.8	26.9
29.0	25.5	25.6	25.7	25.8	25.9	26.1	26.2	26.4	26.6	26.7	26.9	27.1	27.3	27.4
29.5	26.0	26.1	26.2	26.3	26.4	26.6	26.7	26.9	27.1	27.2	27.4	27.6	27.8	27.9
30.0	26.5	26.6	26.7	26.8	26.9	27.1	27.2	27.4	27.6	27.7	27.9	28.1	28.3	28.4
30.5	27.0	27.1	27.2	27.3	27.4	27.6	27.7	27.9	28.1	28.2	28.4	28.6	28.8	28.9
31.0	27.5	27.6	27.7	27.8	27.9	28.1	28.2	28.4	28.6	28.7	28.9	29.1	29.3	29.4
31.5	28.0	28.1	28.2	28.3	28.4	28.6	28.7	28.9	29.1	29.2	29.4	29.6	29.8	29.9
32.0	28.5	28.6	28.7	28.8	28.9	29.1	29.2	29.4	29.6	29.7	29.9	30.1	30.3	30.4
32.5	29.0	29.1	29.2	29.3	29.4	29.6	29.7	29.9	30.1	30.2	30.4	30.6	30.8	30.9
33.0	29.5	29.6	29.7	29.8	29.9	30.1	30.2	30.4	30.6	30.7	30.9	31.1	31.3	31.4
33.5	30.0	30.1	30.2	30.3	30.4	30.6	30.7	30.9	31.1	31.2	31.4	31.6	31.8	31.9
34.0	30.5	30.6	30.7	30.8	30.9	31.1	31.2	31.4	31.6	31.7	31.9	32.1	32.3	32.4
34.5	31.0	31.1	31.2	31.3	31.4	31.6	31.7	31.9	32.1	32.2	32.4	32.6	32.8	32.9
35.0	31.5	31.6	31.7	31.8	31.9	32.1	32.2	32.4	32.6	32.7	32.9	33.1	33.3	33.4

Flash-point, °C.

720	725	730	735	740	745	750	755	760	765	770	775	780	785
17.6	17.8	18.0	18.1	18.3	18.5	18.7	18.8	**19.0**	19.2	19.4	19.5	19.7	19.9
18.1	18.3	18.5	18.6	18.8	19.0	19.2	19.3	**19.5**	19.7	19.9	20.0	20.2	20 4
18.6	18.8	19.0	19.1	19.3	19.5	19.7	19.8	**20.0**	20.2	20.4	20.5	20.7	20.9
19.1	19.3	19.5	19.6	19.8	20.0	20.2	20.3	**20.5**	20.7	20.9	21.0	21.2	21.4
19.6	19.8	20.0	20.1	20.3	20.5	20.7	20.8	**21.0**	21.2	21.4	21.5	21.7	21.9
20.1	20.3	20.5	20.6	20.8	21.0	21.2	21.3	**21.5**	21.7	21.9	22.0	22.2	22.4
20.6	20.8	21.0	21.1	21.3	21.5	21.7	21.8	**22.0**	22.2	22.4	22.5	22.7	22.9
21.1	21.3	21.5	21.6	21.8	22.0	22.2	22.3	**22.5**	22.7	22.9	23.0	23.2	23.4
21.6	21.8	22.0	22.1	22.3	22.5	22.7	22.8	**23.0**	23.2	23.4	23.5	23.7	23.9
22.1	22.3	22.5	22.6	22.8	23.0	23.2	23.3	**23.5**	23.7	23.9	24.0	24.2	24.4
22.6	22.8	23.0	23.1	23.3	23.5	23.7	23.8	**24.0**	24.2	24.4	24.5	24.7	24.9
23.1	23.3	23.5	23.6	23.8	24.0	24.2	24.3	**24.5**	24.7	24.9	25.0	25.2	25.4
23.6	23.8	24.0	24.1	24.3	24.5	24.7	24.8	**25.0**	25.2	25.4	25.5	25.7	25.9
24.1	24.3	24.5	24.6	24.8	25.0	25.2	25.3	**25.5**	25.7	25.9	26.0	26.2	26.4
24.6	24.8	25.0	25.1	25.3	25.5	25.7	25.8	**26.0**	26.2	26.4	26.5	26.7	26.9
25.1	25.3	25.5	25.6	25.8	26.0	26.2	26.3	**26.5**	26.7	26.9	27.0	27.2	27.4
25.6	25.8	26.0	26.1	26.3	26.5	26.7	26.8	**27.0**	27.2	27.4	27.5	27.7	27.9
26.1	26.3	26.5	26.6	26.8	27.0	27.2	27.3	**27.5**	27.7	27.9	28.0	28.2	28.4
26.6	26.8	27.0	27.1	27.3	27.5	27.7	27.8	**28.0**	28.2	28.4	28.5	28.7	28.9
27.1	27.3	27.5	27.6	27.8	28.0	28.2	28.3	**28.5**	28.7	28.9	29.0	29.2	29.4
27.6	27.8	28.0	28.1	28.3	28.5	28.7	28.8	**29.0**	29.2	29.4	29.5	29.7	29.9
28.1	28.3	28.5	28.6	28.8	29.0	29.2	29.3	**29.5**	29.7	29.9	30.0	30.2	30.4
28.6	28.8	29.0	29.1	29.3	29.5	29.7	29.8	**30.0**	30.2	30.4	30.5	30.7	30.9
29.1	29.3	29.5	29.6	29.8	30.0	30.2	30.3	**30.5**	30.7	30.9	31.0	31.2	31.4
29.6	29.8	30.0	30.1	30.3	30.5	30.7	30.8	**31.0**	31.2	31.4	31.5	31.7	31.9
30.1	30.3	30.5	30.6	30.8	31.0	31.2	31.3	**31.5**	31.7	31.9	32.0	32.2	32.4
30.6	30.8	31.0	31.1	31.3	31.5	31.7	31.8	**32.0**	32.2	32.4	32.5	32.7	32.9
31.1	31.3	31.5	31.6	31.8	32.0	32.2	32.3	**32.5**	32.7	32.9	33.0	33.2	33.4
31.6	31.8	32.0	32.1	32.3	32.5	32.7	32.8	**33.0**	33.2	33.4	33.5	33.7	33.9
32.1	32.3	32.5	32.6	32.8	33.0	33.2	33.3	**33.5**	33.7	33.9	34.0	34.2	34.4
32.6	32.8	33.0	33.1	33.3	33.5	33.7	33.8	**34.0**	34.2	34.4	34.5	34.7	34.9
33.1	33.3	33.5	33.6	33.8	34.0	34.2	34.3	**34.5**	34.7	34.9	35.0	35.2	35.4
33.6	33.8	34.0	34.1	34.3	34.5	34.7	34.8	**35.0**	35.2	35.4	35.5	35.7	35.9

[1] "Beilage zum Beglaubigungsschein für den Abelschen Petroleumprober," Physikalisch-Technische Reichsanstalt, **1910.**

Tabulation of Essential Points in State Laws Relating to Illuminating Oil (Kerosene)

State	Cup	Flash °F.	Fire °F.	Gravity	Distillation	Remarks
Alabama	.....	...	120			
Arizona	.....	...	...	.....	...	No law.
Arkansas	Open Tag	...	160			
California	.....	...	...	.....	...	No law.
Colorado	Foster	90				
Connecticut	Open Tag	110	140			
Delaware	Open Tag	...	115			
Florida	.....	...	...	.....	...	No law.
Georgia	Elliott	100				
Idaho	Open Tag	...	120			
Illinois	Open Tag	...	150			
Indiana	Indiana	120	...	50–46		
Iowa	Elliott	100				
Kansas	Foster	110				
Kentucky	Tag	...	130			
Louisiana	Tag	125				
Maine	Tag	...	120			
Maryland	.....	...	...	.....	...	No law.
Massachusetts	Tag	100	110			
Michigan	Foster	120				
Minnesota	Tag	...	120			
Mississippi	.....	...	...	.....	...	No law.
Missouri	Tag	120	...	40 min.	...	4 per cent. residue at 570.
Montana	Foster	110				No law
Nebraska	Foster	112	...	42 min.	...	7 per cent. residue at 570.
Nevada	.....	...	...	.....	...	No law.
New Hampshire	.....	100	120			
New Jersey	Tag	100				
New Mexico	.....	...	120			
New York	Tag	100	110			
North Carolina	Elliott	100	...	.....	...	6 per cent. residue at 570.[1]
North Dakota	Elliott	100	125	.....	...	4 per cent. residue at 570. 6 per cent. max. at 310.
Ohio	Foster	120				
Oklahoma	Tag	115	...	40–48	...	
Oregon	.....	...	...	.....	...	No law.
Pennsylvania	Tag	...	110			
Rhode Island	Tag	...	110			
South Carolina	Elliott	100	....	.....	...	6 per cent. residue at 570.
South Dakota	Elliott	105	...	(47° Pa. crude)	...	Not more than 10 per cent. at 300.
				(41°M-C. do.)	...	Not more than 4 per cent. at 570.
Tennessee	Tag	120				
Texas	.....	...	...	.....	...	No law.
Utah	Foster	110				
Vermont	Tag	...	110			
Virginia	.....	...	...	.....	...	No law.
Washington	Tag electric-cup	...	120			
West Virginia	.....	...	...	.....	...	No law.
Wisconsin	Tag	105	120			
Wyoming	Foster	110	...	.....	...	5 per cent. residue at 572.

[1]North Carolina—If oils are lighter than 47 gravity, then residue must not be more than 10 per cent.

Summary of Government Specifications for Illuminating Oils (Kerosenes)

Oil	Flash	Color	Sulphur	Flock	Distillation	Cloud	Reaction	Burning test	Appearance
Water-white	115°F. or above	21 Saybolt	0.06% or less	None	Distil below 600°F.	No cloud at 0°F.	Neutral	Good flame 18 hrs. without smoke or "ears"	Free from water, glue, or suspended matter
Long-time burning oil	115°F. or above	21 Saybolt		None		No cloud at 0°F.	Neutral	Good flame until consumed or 120 hrs.	Free from water, glue, or suspended matter
300° mineral seal	250°F. or above	16 Saybolt		None		No cloud at 32°F.	Neutral	Must burn until entirely consumed	Free from water, glue, or suspended matter

Flash-points—Tagliabue closed-cup.

Specifications for Illuminating Oil as Adopted by the Railway Signal Association

(*a*) Samples must not flash below a temperature of one hundred and ten (110) degrees Fahr. in the "Elliott" closed-cup nor burn below one hundred and sixty (160) degrees Fahr. in the "Tagliabue" open-cup when heated at the rate of two (2) degrees per minute and flame applied every two (2) degrees, beginning at one hundred (100) degrees Fahr.

(*b*) Must be not less than forty-seven and three-tenths (47.3) Baumé, or a specific gravity of 0.7896.

(*c*) Must be strictly water-white in color and remain perfectly clear and transparent when cooled to a temperature of zero Fahr. and held there for ten (10) minutes. Oil must be perfectly neutral, showing neither acid nor alkaline reaction.

(*d*) A sample will be taken from each carload or part carload lot (either in bulk or in packages) and such sample shall be considered representative of the entire shipment.

(*e*) Samples will be taken with a thief.

(*f*) The oil must burn steadily and clearly in a suitable lamp, without smoking and with a minimum incrustation of the wick for a period of at least one hundred (100) hours.

(*g*) Any sample failing to meet the requirements of these specifications will be condemned and the shipment represented by it will be returned to the manufacturer, who must pay all freight charges.

Specifications for Signal Oil as Adopted by the United States Railroad Administration

METHODS OF TEST

Flash.—To be taken in the Cleveland open-cup. Oil to be heated at the rate of 7°F. per minute and test flame to be applied every 5°, commencing at 210°F.

Fire Test.—After the flash-point is obtained, the oil shall be heated at the same rate (7° per minute). Test flame to be applied every 5° after flash-point has been obtained.

Cloud Test.—Directions for making test: Take a 4-oz. oil sample bottle and introduce therein 1½ oz. of oil to be tested. Insert cork with cold-test thermometer so that bulb is slightly below the surface of the oil. Place the oil in a freezing mixture and cool to 32°F. Keep oil cooled to this temperature for 10 minutes. Bottle should be given a rotary motion occasionally so as not to supercool the sides. The oil should not become cloudy at the end of 10 minutes from crystals of paraffin wax or solid fats from the lard oil or sperm oil.

Burning Test.—This test is to be made in a standard railway signal hand lantern, the burner of which is fitted with a 1-in. wick. The oil to be burned 24 hours without trimming or adjusting the wick, the pot of the lantern to be refilled if too small for a test of the duration named.

Oil must produce a satisfactory flame throughout the test period.

The oil must not produce an appreciable amount of hard incrustation on the wick.

The flame must stand all forms of railroad signaling in any kind of weather without being extinguished or smoking the globe.

SUMMARY OF SPECIFICATIONS—SIGNAL OIL

Appearance.—The oil must be free from water, glue, and suspended matter.

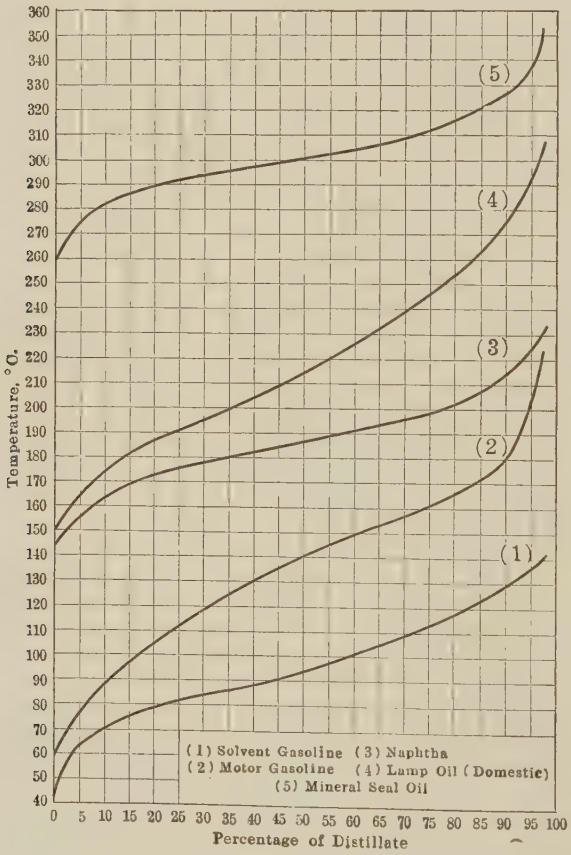


FIG. 121.—Distillation curves of certain petroleum products.

Composition.—To be 300-degree mineral seal oil as adopted by the Committee on Standardization of Petroleum Specifications, compounded with pure prime winter strained lard oil or sperm oil, or compounded with a mixture of pure prime winter strained lard oil and sperm oil.

Flash.—Not less than 250°F. Cleveland open-cup.

Fire.—Not less than 300°F. Cleveland open-cup.

Cloud Test.—Oil should not show cloud at 32°F.

Percentage of Fatty Oil.—"A" grade must contain not less than 30 per cent. of fatty oil by volume. "B" grade must contain not less than 22 per cent. of fatty oil by volume. The "A" grade shall always be furnished unless "B" grade is specifically ordered.

Free Fatty Acids.—"A" grade must contain not over 0.60 per cent. free fatty acid calculated as oleic acid. "B" grade must contain not over 0.45 per cent. free fatty acid calculated as oleic acid.

Burning Test.—As stated above.

Comparison of Saybolt and Lovibond Instruments, with Equivalent Solutions of Potassium Dichromate. (Courtesy of C. K. Francis.)

Potassium dichromate, mg. per liter	Saybolt chromometer	Lovibond colorimeter, 18-in. cell with W.W. slide
2.0	+25	W.W. + 0.5
2.9	+24	W.W.
3.8	+23	W.W. − 0.3
4.7	+22	W.W. − 0.5
5.6	+21	W.W. − 0.8
6.5	+20	W.W. − 1.3
7.5	+19	W.W. − 1.5
8.5	+18	W.W. − 2.0
9.5	+17	W.W. − 2.2
10.5	+16	W.W. − 2.8
11.5	+16	W.W. − 3.0
12.5	+15	W.W. − 3.8
13.5	+14	W.W. − 4.5
14.5	+13	W.W. − 5.2
15.5	+13	W.W. − 5.7
16.5	+12	W.W. − 6.0
17.5	+11	W.W. − 6.5
18.6	+10	W.W. − 6.9
19.7	+ 9	W.W. − 7.4
20.8	+ 8	W.W. − 9.0
21.9	+ 7	W.W. − 9.4
23.0	+ 6	W.W. −10.0
24.1	+ 5	W.W. −10.2
25.3	+ 3	W.W. −11.0
26.6	+ 3	W.W. −11.0
28.0	+ 1	W.W. −11.2

Table of Color Shades for the Saybolt Universal Chromometer

	Inches of oil in tube	Color shades
	20	25
Use one disc.	18	24
	16	23
	14	22
	12	21 = water-white
	10 6/8	20
	9 4/8	19
	8 2/8	18
	7 3/8	17
	6 2/8	16
Use two discs.	10 4/8	+15
	9 6/8	+14
	9 0/8	+13
	8 2/8	+12
	7 6/8	+11
	7 2/8	+10
	6 6/8	+ 9
	6 4/8	+ 8
	6 2/8	+ 7
	6 0/8	+ 6
	5 6/8	+ 5
	5 4/8	+ 4
	5 2/8	+ 3
	5 0/8	+ 2
	4 6/8	+ 1
	4 4/8	0 = standard white
	4 2/8	− 1
	4 0/8	− 2
	3 6/8	− 3
	3 5/8	− 4
	3 4/8	− 5
	3 3/8	− 6
	3 2/8	− 7
	3 1/8	− 8
	3 0/8	− 9

It is evident that no oils are to be compared with one disc unless they positively show whiter at 10 4/8 in. with two discs.

Moreover, a full tube (20 in.) of white oil that shows whiter than one (1) disc must rate +25 and up (better than +25).

Conversion Table for Viscosimeters

(Engler No. 2204 U and Standard Saybolt Universal)

Time, Engler, sec.	Time, Saybolt, sec.	Time, ratio	Engler degrees = time, Engler / 51.3[a]	Kinematic viscosity	Time, Engler, sec.	Time, Saybolt, sec.	Time, ratio	Engler degrees = time, Engler / 51.3[a]	Kinematic viscosity, Poises, g/cm.³
56	32.4	1.73	1.09	0.0155	130	83.6	1.56	2.54	0.1624
58	33.7	1.72	1.13	0.0208	140	90.6	1.55	2.73	0.1793
60	35.1	1.71	1.17	0.0259	150	97.4	1.54	2.93	0.1956
62	36.5	1.70	1.21	0.0308	160	104.4	1.53	3.12	0.2121
64	37.8	1.69	1.25	0.0356	180	117.7	1.53	3.51	0.2437
66	39.2	1.68	1.29	0.0403	200	131.5	1.52	3.90	0.2753
68	40.6	1.68	1.33	0.0448	225	148.3	1.52	4.39	0.3140
70	42.0	1.67	1.36	0.0495	250	165.3	1.51	4.88	0.3523
75	45.5	1.65	1.46	0.0603	275	182.0	1.51	5.36	0.3904
80	49.0	1.63	1.56	0.0709	300	199.0	1.51	5.85	0.4282
85	52.4	1.62	1.66	0.0810	325	215.6	1.51	6.34	0.4660
90	55.8	1.61	1.76	0.0905	350	232.9	1.50	6.82	0.5038
95	59.4	1.60	1.85	0.1003	375	249.2	1.50	7.31	0.5413
100	62.8	1.59	1.95	0.1095	400	266.1	1.50	7.80	0.5874
110	69.8	1.58	2.15	0.1278	500	333.0	1.50	9.75	0.7271
120	76.8	1.56	2.34	0.1453	600	400.0	[b]1.50	11.70	0.8753

[a] Bureau of Standards *Tech. Paper* **100.**

[b] This value holds good for all higher viscosities.

Conversion Table for Redwood, Saybolt, and Engler Viscosimeters

Time, Engler, sec.	Time, ratio			Time, Redwood, sec.	Time, Engler, sec.	Time, ratio			Time, Redwood,
	Engler-Saybolt	Engler-Redwood	Saybolt-Redwood[a]			Engler-Saybolt	Engler-Redwood	Saybolt-Redwood[a]	
56	1.73	2.61	1.51	21.5	140	1.55	1.81	1.17	77.5
58	1.72	1.93	1.12	30.0	150	1.54	1.80	1.17	83.2
60	1.71	1.93	1.13	31.2	160	1.53	1.80	1.17	89.1
62	1.70	1.92	1.13	32.2	180	1.53	1.80	1.17	100.3
64	1.69	1.91	1.13	33.4	200	1.52	1.79	1.18	111.9
66	1.68	1.91	1.13	34.6	225	1.52	1.79	1.18	126.0
68	1.68	1.90	1.14	35.8	250	1.51	1.78	1.18	140.3
70	1.67	1.90	1.14	36.9	275	1.51	1.78	1.18	154.4
75	1.65	1.88	1.14	39.8	300	1.51	1.78	1.18	168.5
80	1.63	1.87	1.15	42.7	325	1.51	1.78	1.18	183.0
85	1.62	1.86	1.15	45.7	350	1.50	1.78	1.18	197.0
90	1.61	1.86	1.15	48.5	375	1.50	1.77	1.18	211.3
95	1.60	1.85	1.16	51.4	400	1.50	1.77	1.18	225.5
100	1.59	1.84	1.16	54.3	500	1.50	1.77	1.18	282.0
110	1.58	1.83	1.16	60.1	600	1.50	1.77	1.18	339.0
120	1.56	1.82	1.16	66.0					
130	1.56	1.81	1.17	71.7					

[a] These values were calculated directly from the times of discharge and differ in some cases by 0.01 from values calculated from the two previous columns.

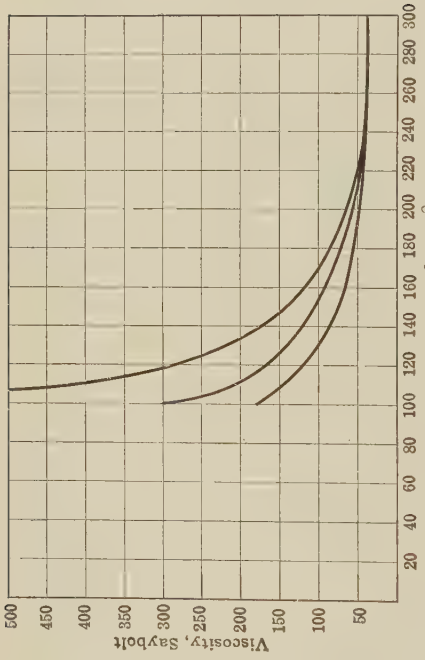


FIG. 122.—Curves illustrating the effect of temperature on viscosity (Bryan, *J. Am. Soc. Naval Eng.*, Nov., **1915**).

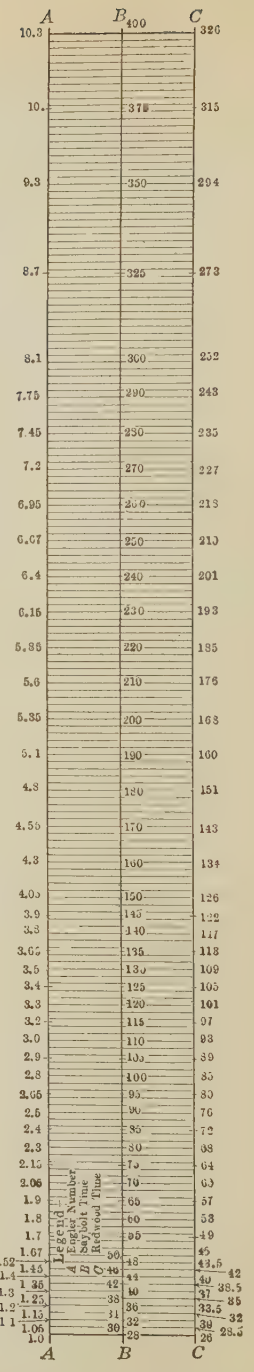


FIG. 123.—Viscosity transition chart (Fred Carl), not based upon the conversion tables given, but upon older data.

A Practical Method for Calculating the Viscosity of Hydrocarbon Oil Mixtures

By W. E. Espy[1]

It is a well-known fact that, in mixing oils having different viscosity numbers, the resulting viscosity does not correspond with the calculated viscosity for such a blend, but that the observed viscosity will be lower. For example, a mixture composed of 50 per cent. 100 viscosity oil and 50 per cent. 300 viscosity oil, should result, theoretically, in a blend having 200 viscosity. The actual determined viscosity of this blend will, however, be approximately 165, or 82.5 per cent. of the calculated figure.

It has been shown by Sherman, Gray and Hammerschlag,[2] in their work on this subject, that a graph drawn from mixtures of fatty oils with mineral oils does not correspond to mixtures of mineral oil only.

Work in this laboratory has demonstrated that a certain amount of fatty oil will have a greater effect in raising or lowering the viscosity of a mixture with mineral oil, than the same amount of mineral oil. For this reason the desired viscosity of fatty and mineral oil mixtures cannot be calculated from the method to follow.

To avoid empirical trial, F. Schulz[3] has experimentally determined the constants, with the aid of which it is possible to calculate the percentage of each of the oils necessary for the mixture. For mixtures of 5 to 50 per cent. of the more viscous oil the viscosity sought is:

$$G = (0.166^n)(V) + 1.161\frac{(1 - 0.166^n)}{(1 - 0.166)}(v);$$

and for mixtures of 5 to 50 per cent. of the less viscous oil:

$$G = (1.161^n)(v) + 0.166\frac{(1.161^n - 1)}{(1.161 - 1)}(V).$$

Here V represents the viscosity of the more viscous, and v that of the less viscous oil.

The exponent n is calculated by the formula:

$$n = \frac{\log p - \log 100}{\log 0.5},$$

where p is the volume percentage of one of the components.

From the equations given for mixtures of from 5 to 95 per cent. of the more viscous oil, the viscosity in Engler degrees can be calculated by the following formulas:

$$G_5 = v\left(1.392 + 0.0004\frac{V}{v}\right) \qquad G_{30} = v\left(1.331 + 0.044\frac{V}{v}\right)$$

$$G_{10} = v\left(1.388 + 0.0026\frac{V}{v}\right) \qquad G_{35} = v\left(1.300 + 0.066\frac{V}{v}\right)$$

$$G_{15} = v\left(1.382 + 0.007\frac{V}{v}\right) \qquad G_{40} = v\left(1.262 + 0.093\frac{V}{v}\right)$$

$$G_{20} = v\left(1.371 + 0.015\frac{V}{v}\right) \qquad G_{45} = v\left(1.217 + 0.126\frac{V}{v}\right)$$

$$G_{25} = v\left(1.354 + 0.028\frac{V}{v}\right) \qquad G_{50} = v\left(1.161 + 0.166\frac{V}{v}\right)$$

[1] Private communication from Cosden and Company, Tulsa, Okla.

[2] *J. Ind. Eng. Chem.*, **1** (1909), 13.

[3] "The Examination of Hydrocarbon Oils," by Holde-Mueller, 111.

$$G_{55} = v\left(1.187 + 0.193\frac{V}{v}\right) \qquad G_{80} = v\left(1.414 + 0.427\frac{V}{v}\right)$$

$$G_{60} = v\left(1.218 + 0.225\frac{V}{v}\right) \qquad G_{85} = v\left(1.505 + 0.521\frac{V}{v}\right)$$

$$G_{65} = v\left(1.254 + 0.262\frac{V}{v}\right) \qquad G_{90} = v\left(1.642 + 0.662\frac{V}{v}\right)$$

$$G_{70} = v\left(1.296 + 0.305\frac{V}{v}\right) \qquad G_{95} = v\left(1.906 + 0.934\frac{V}{v}\right)$$

$$G_{75} = v\left(1.348 + 0.359\frac{V}{v}\right)$$

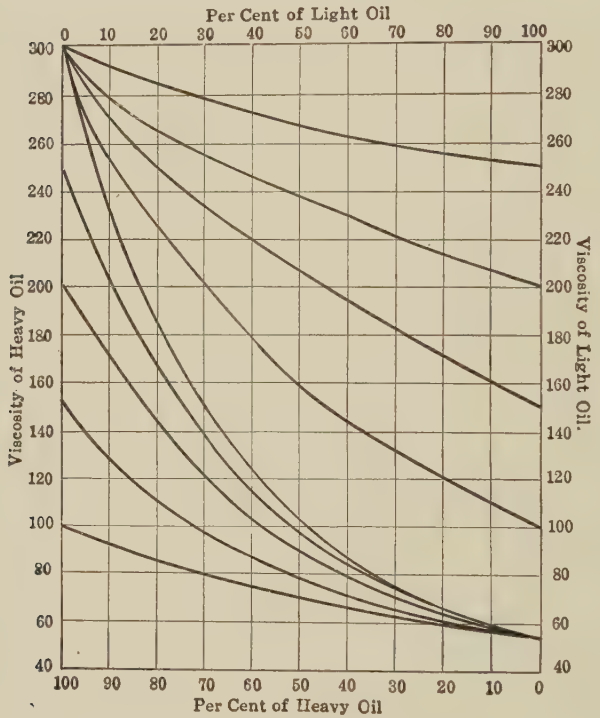


FIG. 124.—Saybolt viscosity mixtures.

In these formulas, G_5, for example, means the Engler degree with 5 per cent. by volume of the more viscous oil.

The Engler values thus calculated agree with the experimental to within one unit; only when the mixtures contain less than 10 per cent., or more than 90 per cent., may the error be more than fifteen units.

This formula can be used as a guide in mixing for viscosity, but is rather cumbersome and would require several trial calculations before arriving at the desired result.

It is the object of this report to illustrate a quick, simple method for

calculating viscosity mixtures, which will be interesting to those connected with work of this nature.

EXPERIMENTS

Two graphic charts are given herewith, the curves having been plotted to show the deviation from the theoretical figures, which would, of course, be indicated by the intersection of a straight line between the terminals of the curves shown.

Fig. 124 illustrates the result of several mixtures of oil manufactured from Mid-Continent crude, the viscosities thereof being widely different, while others are more nearly alike.

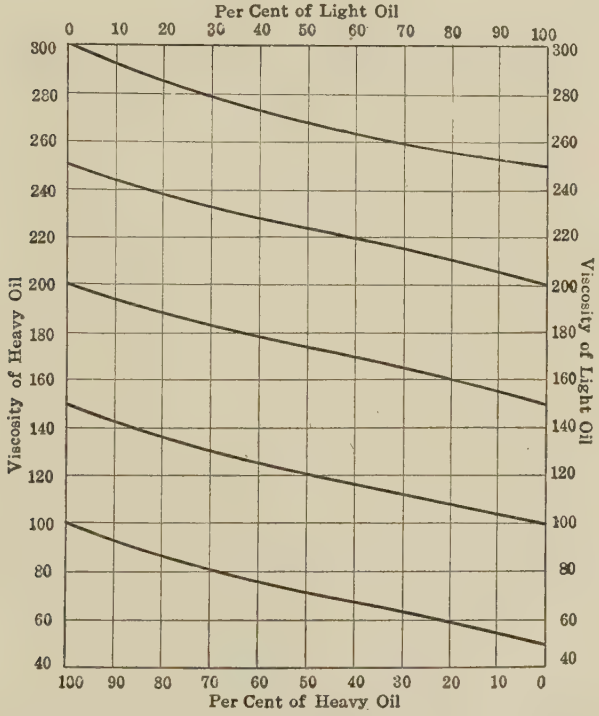


Fig. 125.—Saybolt viscosity mixtures.

In Fig. 125 the oils mixed have a difference of 50 points, and range from 55 to 300.

In order to ascertain if there is any variation in the contour of curves plotted from mixtures of oils obtained from the same crude, and those from a crude of totally different character, such as Pennsylvanian and Mexican, the following mixtures were made:

VISCOSITY BLENDS, SHOWING VARIOUS MIXTURES OF 55 AND 300 VISCOSITY

Heavy oil, kind Light oil, kind Temperature Viscosimeter		Okla. Okla. 100°F. Saybolt	Okla. Okla. 210°F. Saybolt	Texas Okla. 100°F. Saybolt	Okla. Okla. 100°F. Engler	Calculated
Per cent. light	Per cent. heavy					
100	0	55	55	55	55	55
75	25	71	72	72	59	116
50	50	103	104	102	63	178
25	75	167	166	165	108	239
0	100	300	300	300	300	300

The two oils used in the above table, namely, lubricating oils from Goose Creek (Texas) crude and Cushing (Okla.) crude, are widely divergent in character, but the results obtained so nearly coincide with those shown in the table which follows and other different mixtures, that the results of either would be close enough for practical purposes.

CALCULATION

While viscosity numbers are not additive figures, they correspond to certain percentage mixtures of lighter and heavier oils than those on which the calculation is to be based.

For example, an oil having a viscosity of 500 units is desired from a mixture of 700 and 300 viscosity. Each of these three figures could be represented by a certain percentage of 50 viscosity oil with 1000 viscosity oil.

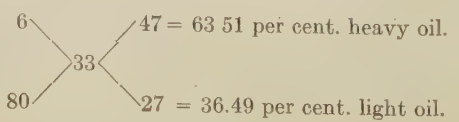
To illustrate this point better, the figures in the following table were determined by experiment, viscosities between 55 and 300 Saybolt being used, as their application is more general for commercial purposes.

Taking a concrete case, a mixture having 140 viscosity is desired from two oils of 249 and 68 viscosity, respectively. Instead of using these figures, which would give by direct calculation a high percentage of low viscosity oil to be used, we use figures representing percentage of lower viscosity oil with a higher viscosity oil, necessary to give the above mentioned viscosities.

140 viscosity Saybolt equals 33 per cent.
249 viscosity Saybolt equals 6 per cent.
68 viscosity Saybolt equals 80 per cent.

If we accept the oil as being a certain percentage solution, the percentage representing a certain viscosity may be used as an additive figure for calculating viscosity mixtures.

The example given may be solved by a simple calculation, using the rectangular formula, thus:



SAYBOLT VISCOSITY

(Per cent. given equals amount of 55 viscosity oil mixed with 300 viscosity oil)

Per cent.	Vis.	Per cent.	Vis.	Per cent.	Vis.	Per cent.	Vis.	Per cent.	Vis.
100	55								
99	55	79	68	59	88	39	124	19	189
98	55	78	69	58	89	38	127	18	193
97	56	77	70	57	91	37	129	17	197
96	56	76	71	56	93	36	132	16	201
95	57	75	72	55	95	35	135	15	205
94	57	74	72	54	96	34	137	14	209
93	58	73	73	53	98	33	140	13	214
92	58	72	74	52	99	32	143	12	219
91	59	71	75	51	101	31	146	11	224
90	60	70	76	50	103	30	149	10	229
89	60	69	77	49	104	29	152	9	234
88	61	68	78	48	106	28	156	8	239
87	62	67	79	47	108	27	159	7	244
86	63	66	80	46	110	26	163	6	249
85	64	65	81	45	112	25	167	5	254
84	64	64	82	44	114	24	170	4	261
83	65	63	83	43	116	23	174	3	269
82	66	62	84	42	118	22	177	2	278
81	67	61	86	41	120	21	181	1	288
80	68	60	87	40	122	20	185	0	300

The viscosity of a blend, calculated by the above method, will check within a limit of three points. It would be necessary to interpolate figures not given in the preceding table.

The figures presented would not hold true for Engler or other instruments, as another chart would have to be prepared for them.

Provided the viscosities of the oils to be blended and the resulting mixture are determined at the same temperature, the figures in the second table can be used at any temperature.

CHART FOR CALCULATION

A much better method is the arrangement of a chart for calculating viscosity mixtures. This may be accomplished in a simple manner, as follows:

Make solutions of a low viscosity oil in a high viscosity oil at each 1 per cent. or fractions of 1 per cent. from 1 to 99.

Make a chart using the viscosities of these mixtures as abscissæ, placing the lower figures at the bottom on each side, and the higher figures at the top, rising in consecutive order, spaced at equal distances for each 1 per cent.

Percentages from 1 to 99 are used as ordinates, the figures at the top reading from right to left, and those at the bottom from left to right.

A convenient size for industrial use is 20 × 20 in.

MANIPULATION

Connect the two given viscosities by means of a straight edge. Reading up from the required viscosity line at the point of intersection, the percentage of light liquid will be found, and reading down, the percentage of heavy liquid to make the desired product will be secured.

Example.—A product of 112 viscosity is desired from 67 viscosity and 299 viscosity. A blend of 50 per cent. light and 50 per cent. heavy oil should be used.

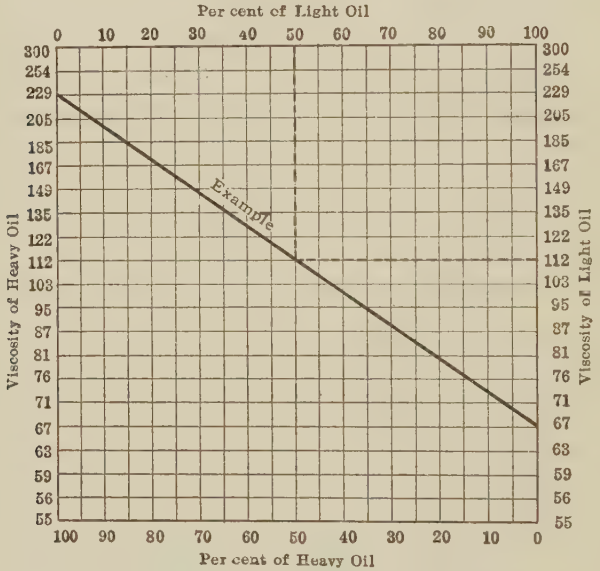


FIG. 126.—Viscosity blending chart for Saybolt viscosity.

Space will not permit an illustration of a chart large enough for accurate work. Figure 126, being an example, shows figures only at each 5 per cent.

A chart 20 in. square permits the viscosity at each additional 1 per cent. to be shown; in this case a 1 per cent. addition of heavy oil to the light makes a difference of less than one second, while the addition of 1 per cent. light oil to the heavy oil makes a difference of 12 seconds.

This necessitates the interpolation of figures which cannot be given between the lines, at the top of the chart.

SUMMARY

1. Viscosity numbers are not additive figures, and there is no set rule or factor applicable to the calculation of viscosity mixtures, which will account for the greater influence of the lower viscosity liquid used.

2. Co-ordinate curves are obtained by the mixture of hydrocarbon oils of different sources and properties, when using the same type viscosimeter in all cases.

3. Similar curves are also obtained at different temperatures for similar viscosities with the same instrument, provided the temperatures are not low enough to cause a partial crystallization of the liquid.

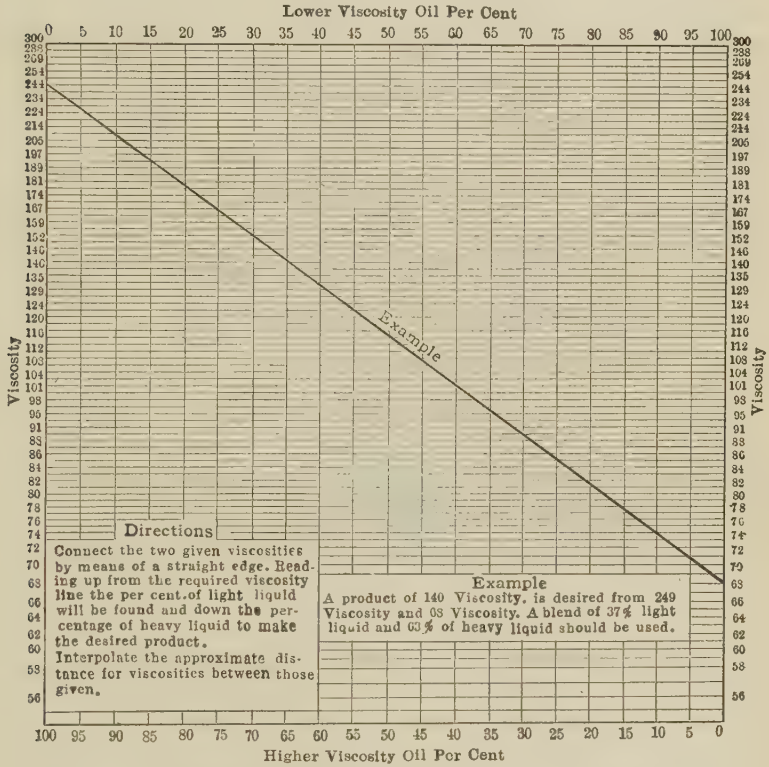

Lower Viscosity Oil Per Cent
0 5 10 15 20 25 30 35 40 45 50 55 60 65 70 75 80 85 90 95 100
Viscosity
300 288 269 254 244 234 224 214 205 197 189 181 174 167 159 152 146 140 135 129 124 120 116 112 108 104 101 98 95 91 88 86 84 82 80 78 76 74 72 70 68 66 64 62 60 58 56
Example
Directions
Connect the two given viscosities by means of a straight edge. Reading up from the required viscosity line the per cent. of light liquid will be found and down the percentage of heavy liquid to make the desired product. Interpolate the approximate distance for viscosities between those given.
Example
A product of 140 Viscosity, is desired from 249 Viscosity and 93 Viscosity. A blend of 37% light liquid and 63% of heavy liquid should be used.
100 95 90 85 80 75 70 65 60 55 50 45 40 35 30 25 20 15 10 5 0
Higher Viscosity Oil Per Cent

FIG. 127.—The viscosity blending chart of W. E. Espy. Reference is had to Saybolt viscosities.

4. Any admixture of fatty oils with mineral oils will cause a distortion of the curves given; a lower result being obtained when the mineral oil has a higher viscosity, and a higher result when the mineral oil is of lower viscosity.

5. Any viscosity number may be accepted as a percentage mixture of higher and lower viscosities.

These percentages may be used as additive figures in calculation, after determining the corresponding viscosity by experiment. A chart arranged for viscosity calculation by the above method can be used for any temperature, but a different chart is necessary for each type of viscosimeter.

The Saybolt Viscosity of Blends

By Winslow H. Herschel,

U. S. Bureau of Standards.

It has long been recognized that the viscosity of a mixture or blend of two oils is less than the arithmetical mean of the viscosities of the component oils, whether expressed in Saybolt seconds, poises, or other unit of viscosity.

The viscosity of liquid mixtures has been discussed by Dunstan and Thole,[1] who give a formula which applies with considerable accuracy to definite chemical compounds which are inert with respect to each other. This formula is based on molecular weights and can not be used to estimate the viscosity of blends of petroleum oils which are not definite chemical compounds.

A convenient diagram for determining the viscosity of blends has been given by Espy[2] for viscosities between 300 and 55 seconds, Saybolt, but no equation is presented by which the diagram may be extended beyond the range of his experiments. For more general use, the best available formula is that of Arrhenius:[3]

$$\log \mu = v_1 \log \mu_1 + v_2 \log \mu_2, \tag{1}$$

where v_1 and v_2 are the percentage volume concentrations, and μ_1 and μ_2 are the viscosities of the component oils, expressed in poises. Otherwise expressed, equation (1) gives the viscosity of a blend as the weighted geometrical mean between the viscosities of the component oils, so that the logarithms of the viscosities, in poises, are additive. It has been found, however, from the tests here recorded, that the values calculated from equation (1) may be considerably larger than the experimentally determined values, when there is a large difference of viscosity between the component oils.

CALCULATED TABLE OF VISCOSITIES OF BLENDS

When the Saybolt universal viscosimeter is used, the viscosity μ, in poises, may be calculated by the equation,

$$\mu = (.00220\, t - \frac{1.80}{t})\gamma, \tag{2}$$

where t is the time of flow, or Saybolt viscosity, in seconds, and γ is the density of the oil at the temperature of test, in grams per cubic centimeter.[4]

To avoid at least part of the waste of time and labor involved in cut and try methods, it is desirable to have available a simple table or diagram, as accurate as possible, to solve the problem of obtaining a blend of desired

[1] A. E. Dunstan and F. B. Thole, "The Viscosity of Liquids," **1914**, 39–51.

[2] W. E. Espy, *Petroleum*, **8** (1919), 27. See also page 362.

[3] S. Arrhenius, *Z. physikal. Chem.*, **1** (1887), 285.

[4] Herschel, *Proc. Am. Soc. Testing Materials*, **18** (1918), ii, 363; *Tech. Paper* No. **112**, Bureau of Standards.

viscosity from two oils of known viscosity. Since there is no definite relation between the viscosity and the density of oils (except when they are all from the same crude), it is necessary in calculating such a table to assume for the sake of simplicity that the two component oils have the same density. Then values of $\frac{\mu}{\gamma}$ obtained from equation (2) may be used in equation (1) in place of μ without affecting its validity, and the resulting values of $\frac{\mu}{\gamma}$ of the blend may be converted into corresponding Saybolt viscosities by means of the equation:

$$t = 227.3\frac{\mu}{\gamma}\left(1 + \sqrt{1 + \frac{.01584}{\left(\frac{\mu}{\gamma}\right)^2}}\right) \qquad (3)$$

Table I shows the Saybolt viscosities of blends containing 25, 50 and 75 per cent. of either of the component oils. It was calculated from equations (1) and (2), the need of the somewhat more troublesome equation (3) being avoided by a graphical method. Each line of the table shows values of Saybolt viscosity for blends with a given viscosity of the lighter oil, the headings of the columns showing the viscosity of the heavier oil. Each rectangle formed by the intersection of a line and a column contains three values which are viscosities of blends of the two oils in question. Beginning at the top of a rectangle these values are for blends containing 25, 50 and 75 per cent., respectively, of the heavier oil.

Somewhat more accurate results may be obtained if correction is made for the difference in density between the component oils. If the last term in equation (2) is negligible, and it is assumed that the table applies to oils of unit density, the correction for density may be made by entering the table with the corrected Saybolt viscosity t_γ. Correcting the viscosities of both components in this manner, and finding from the table (or from a similar table for higher viscosities) the uncorrected viscosity of the blend, T, it is necessary first to find the density of the blend ρ by taking the weighted arithmetical mean of the densities of its components. Then the desired viscosity of the blend is equal to $\frac{T}{\rho}$.

BLENDING CHARTS

On account of the large number of variables, it is difficult to present Table I graphically. Any pair of oils may be selected, as in Fig. 128, but to give all the information in the table in this manner would necessitate a large number of diagrams with no means of readily interpolating between them. If Saybolt viscosities were additive the lines of Fig. 128 would be straight, but, as may be seen, the greater the difference in the viscosity of the component oils, the greater the curvature of the lines.

Figure 129 is perhaps the most satisfactory form of diagram for a blending chart. Its form is due to Espy, but it was plotted from data of Table I. According to Espy, the data from a series of tests of two component oils mixed in all proportions are sufficient to enable a chart to be prepared

which will be applicable to oils of all crudes, but, as will be shown later, tests covering a wider range of viscosities do not confirm this.

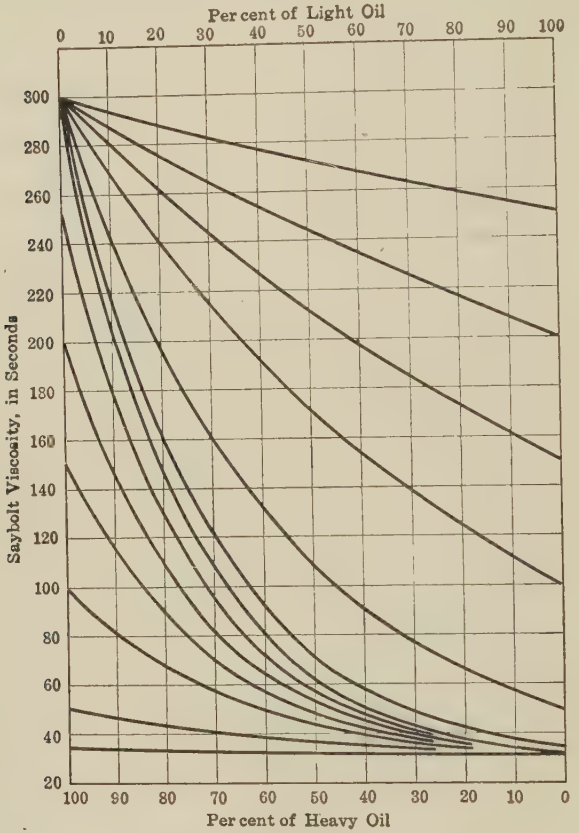


FIG. 128.

CHECK OF ESPY'S RESULTS

While there was a fair agreement between Espy's results and Table I, it was desired to determine whether the differences which appeared were due to differences in viscosimeters, the neglect of density variations in calculating the table, or other causes. For this purpose, the tests of this series were made with two oils of approximately the same viscosities as used by Espy. The specific gravities of these oils were 0.850 for the light and 0.878 for the heavy, while Espy's values[1] were 0.872 and 0.903, respectively.

If μ is the viscosity of a blend as calculated from equation (2) and μ' is the viscosity, in poises, as estimated from the viscosities of the two com-

[1] Private communication.

Table I

Saybolt Viscosities of Blends Containing 25, 50 and 75 Per Cent. of Heavier Oil, Both Constituents Being at the Same Temperature as the Blend

Viscosity of heavier oil	35	40	45	50	60	70	80	90	100	110	120	130	140	150	160	170	180	190	200	210	220	230	240	250	260	270	280	290	300	400	
	33 33 34	33 35 37	34 36 40	34 37 42	35 39 46	35 40 50	35 41 54	36 43 58	36 44 62	36 45 65	36 46 68	37 47 72	37 47 75	37 48 78	37 49 81	37 50 84	37 51 88	38 52 91	38 53 93	38 53 96	38 54 99	38 55 103	38 56 106	38 57 109	39 57 112	39 58 114	39 59 117	39 60 120	39 61 123	40 65 150	32
300	322 347 372		36 37 38	37 39 42	37 40 44	38 42 49	39 44 54	39 46 58	40 48 63	40 49 67	40 51 71	41 52 75	41 54 79	41 55 83	42 56 87	42 57 91	42 58 94	43 59 98	43 60 101	43 61 105	43 62 108	43 63 111	43 64 115	43 65 118	44 66 122	44 67 125	44 68 128	44 69 131	44 70 135	45 71 138	46 79 170	35
290	314 341 369	293 295 297		41 42 43	42 44 47	42 47 52	44 49 58	45 52 63	45 55 68	46 57 73	47 59 78	47 60 82	48 62 87	48 63 91	48 65 95	49 67 100	50 69 105	50 70 109	50 72 113	51 73 117	51 74 121	51 75 125	51 77 129	52 78 133	52 79 137	52 80 140	53 81 144	53 83 148	53 84 152	54 85 155	55 96 191	40
280	305 334 366	285 290 294	282 285 287		46 47 49	48 52 56	49 55 61	50 58 67	52 61 73	52 63 78	53 65 83	54 67 88	55 69 93	56 71 98	57 73 102	57 75 107	58 77 112	58 79 118	59 81 122	59 83 126	60 85 130	60 86 135	60 88 139	61 90 143	61 91 148	62 93 152	62 94 156	63 96 160	63 97 165	64 99 169	66 112 208	45
270	297 328 362	277 283 290	274 279 284	272 275 277		52 55 57	54 59 64	56 62 70	57 65 76	58 68 82	59 71 87	60 73 92	61 76 98	62 78 103	63 80 108	64 82 113	64 85 118	65 87 124	65 89 128	66 91 132	66 93 137	67 95 142	67 97 147	68 99 151	68 100 155	69 102 160	69 104 164	70 105 168	70 107 173	71 108 177	74 124 220	50
260	289 322 359	272 279 289	269 274 282	266 270 275	263 265 267		62 64 67	63 68 74	65 72 80	66 76 87	68 79 93	69 81 99	70 84 105	71 87 110	72 90 115	73 93 120	74 96 126	75 98 131	75 100 136	76 102 142	77 104 147	77 106 152	78 108 157	79 110 162	80 113 166	80 115 171	81 117 176	81 119 181	82 121 186	82 123 191	87 141 235	60
250	280 316 355	261 273 285	259 269 278	257 264 271	255 260 264	252 255 257		72 74 77	74 79 84	75 83 91	77 86 98	78 89 102	80 93 109	81 96 115	82 99 121	83 102 127	84 105 133	85 108 139	86 111 144	88 113 150	89 116 155	90 119 160	91 121 165	92 124 170	92 126 176	93 129 182	94 131 187	94 133 192	95 135 198	96 138 202	102 157 249	70
240	272 309 352	253 268 283	251 263 276	249 259 269	247 254 262	244 250 255	242 245 247		82 85 87	84 89 95	86 93 101	87 96 106	89 101 115	91 104 120	92 108 126	94 111 133	95 114 139	97 118 144	98 121 150	99 124 156	100 126 162	101 129 168	102 132 173	102 135 179	103 138 185	104 140 190	105 143 196	106 145 201	106 148 206	107 150 212	115 171 260	80
230	264 302 348	245 263 280	243 259 273	241 254 266	239 250 259	237 245 252	235 240 245	233 235 238		93 95 97	95 100 105	96 103 111	98 107 118	100 111 124	101 115 132	103 119 139	105 122 145	106 126 150	108 129 156	109 132 162	110 135 168	111 139 174	112 141 180	113 144 186	114 147 192	115 149 197	116 152 202	117 155 208	118 157 213	119 160 219	128 184 271	90
220	255 296 344	237 257 276	236 252 270	234 248 262	232 244 256	229 239 249	227 235 242	225 230 235	222 225 227		103 104 108	105 109 115	107 114 122	109 118 129	110 122 135	112 126 142	114 130 148	115 133 155	117 137 161	118 140 168	120 143 174	121 146 180	122 150 186	123 153 192	125 156 198	126 159 204	127 162 209	128 165 215	129 168 220	131 171 226	139 196 279	100
210	246 289 340	229 251 274	227 247 267	225 243 260	224 239 253	221 234 246	219 229 239	217 225 232	215 220 225	212 215 217		112 114 117	114 119 124	116 124 131	118 128 138	120 132 145	122 136 151	124 140 158	125 143 165	127 147 171	129 150 178	130 153 184	131 157 190	133 160 196	134 164 202	135 167 208	137 170 214	138 173 220	139 177 226	140 180 232	149 206 287	110
200	237 282 335	221 245 270	219 241 263	218 237 257	216 233 250	214 228 243	211 224 236	209 219 229	207 215 222	205 210 215	202 205 207		121 124 128	124 129 135	126 134 142	129 138 149	131 142 156	133 146 162	135 150 169	136 153 176	138 157 182	140 160 188	141 164 195	142 168 201	144 171 207	145 175 213	146 178 219	147 182 225	148 185 231	149 188 237	160 215 293	120
190	228 274 331	213 238 266	211 234 260	209 230 253	208 226 246	206 222 240	204 218 233	201 213 226	199 208 219	197 204 212	195 199 205	192 195 197		132 135 138	134 139 145	136 143 152	138 147 159	140 152 165	142 156 172	144 160 179	145 164 179	147 168 185	148 172 192	150 176 199	151 179 205	153 183 211	154 186 218	155 190 224	157 193 230	158 196 236	170 225 242	130
180	219 267 326	204 231 263	202 228 257	200 224 250	199 220 244	197 216 237	195 212 230	193 208 223	191 203 216	189 199 208	187 194 201	185 190 194	182 185 187		142 144 147	145 149 154	147 154 161	149 158 168	150 162 175	152 167 182	154 171 189	155 175 195	157 179 202	158 182 209	160 186 215	161 190 222	163 194 228	164 197 234	166 200 241	167 204 247	180 234 306	140
170	210 259 321	195 225 260	194 222 254	192 218 247	190 214 241	189 210 234	187 206 227	185 202 220	183 198 213	181 193 206	179 189 199	177 184 192	175 180 185	172 175 177		152 155 157	155 159 165	157 164 172	159 169 179	161 173 186	163 178 193	165 182 200	167 186 207	168 190 213	170 193 220	172 197 226	173 200 232	175 204 239	176 207 245	178 211 252	190 244 311	150
160	200 252 316	187 218 256	186 215 250	184 211 244	183 208 237	181 204 231	179 200 224	177 196 217	175 192 210	174 188 204	172 183 197	170 179 190	167 174 183	165 170 175	163 165 168		163 165 168	165 170 175	167 174 183	170 179 190	172 183 197	174 188 204	175 192 210	177 196 217	179 200 224	181 201 231	183 208 237	184 211 244	186 215 250	187 218 256	200 252 316	160
	400	300	290	280	270	260	250	240	230	220	210	200	190	180	170		170	180	190	200	210	220	230	240	250	260	270	280	290	300	400	

Viscosities of lighter oil over 160 vis.

Viscosities of lighter oil less than 160 vis.

Viscosities of heavier oil, for lighter oils *over* 160 vis. ←

→ Viscosities of heavier oil, for lighter oils *less than* 160 vis.

NOTE.—The 3 values in each rectangle are viscosities of blends of the 3 percentage compositions given above.

(*Facing page* 366)

[4] Private communication.

ponents, then $\frac{\mu}{\mu'}$ is a correction factor by which viscosities, estimated by equation (1), must be multiplied to obtain the values as given by test. $\frac{t'}{t}$ is a corresponding correction factor for Saybolt viscosities.

Table II shows correction factors calculated from results of tests of this series. It will be noticed that there is considerable difference between

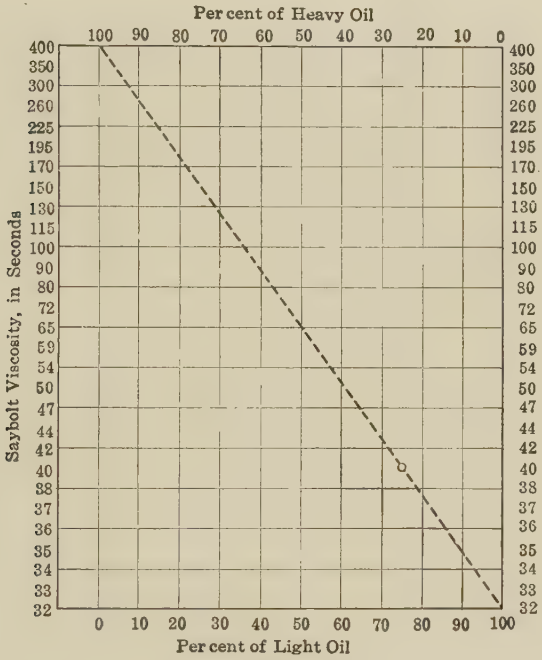


Fig. 129.

Note.—As shown by the point on the dotted line, a blend of 40 viscosity may be made from 25 per cent. of oil of 400 viscosity and 75 per cent. of oil of 32 viscosity, both oils and the blend being at the same temperature.

the correction factors for viscosities in poises and in Saybolt seconds, for a given test at low viscosities, but that the difference becomes negligible at high viscosities. The table confirms in a general way Espy's results for a limited range of viscosities, and shows that there is a systematic error in equation (1). Table I is affected by this error, as well as by the neglect of variations of density in its calculation.

TABLE II.—CHECK OF ESPY'S RESULTS

Per cent. of lighter oil in blend	W. E. Espy tests at 100°F. (37.8°C.)				Bureau of Standards tests at 68°F. (20°C.)			
	t	μ	$\frac{t}{t'}$	$\frac{\mu}{\mu'}$	t	μ	$\frac{t}{t'}$	$\frac{\mu}{\mu'}$
100	55	0.0754	.....	.....	56.2	0.0775		
95	57	0.0804	0.977	0.964	60.1	0.0866	1.005	1.012
90	60	0.0876	0.967	0.948	63.1	0.0936	0.994	0.991
85	64	0.0968	0.965	0.944	66.0	0.1002	0.974	0.961
80	68	0.1060	0.955	0.934	70.1	0.1094	0.971	0.951
75	72	0.1153	0.937	0.918	74.8	0.1198	0.954	0.940
70	76	0.1240	0.916	0.893	80.2	0.1316	0.951	0.937
65	81	0.1351	0.902	0.878	85.2	0.1424	0.933	0.919
60	87	0.1483	0.892	0.870	90.4	0.1534	0.913	0.897
55	95	0.1653	0.895	0.876	98.2	0.1698	0.911	0.898
50	103	0.1823	0.889	0.872	107.8	0.1895	0.920	0.909
45	112	0.2014	0.886	0.869	114.6	0.2037	0.897	0.884
40	122	0.2220	0.880	0.866	124.5	0.2239	0.891	0.881
35	135	0.2486	0.886	0.876	141.5	0.2580	0.924	0.921
30	149	0.2776	0.890	0.882	154.5	0.2840	0.922	0.917
25	167	0.3139	0.907	0.901	169.2	0.3138	0.921	0.917
20	185	0.3502	0.914	0.908	187.0	0.3490	0.926	0.923
15	205	0.3909	0.918	0.916	203.1	0.3813	0.916	0.915
10	229	0.4390	0.932	0.927	240.4	0.4540	0.989	0.985
5	254	0.4895	0.936	0.935	267.3	0.507	0.996	0.995
0	300	0.580	.....	.....	295.2	0.562	.....	.....

TESTS WITH OILS FROM DIFFERENT CRUDES, BUT OF APPROXIMATELY THE SAME VISCOSITY AT THE TEMPERATURE OF TEST

In order to determine whether the correction factor varies with the source of the crude oil, two heavy oils, N and P, one of naphthene and the other of paraffin crude, were selected of approximately the same viscosity at 55°C. (131°F.) and blended with two lighter oils M and Q of the same crudes. With a time of flow much over 500 seconds it is very difficult to get check results; but if the time is decreased by raising the temperature, there is an increase in the error due to the change in volume of the oil after it leaves the outlet tube. The temperature of 55°C. was adopted as a compromise between these considerations.

It was found that the maximum correction factor was obtained for a blend of N and Q and the minimum factor for P and M, and it is noteworthy that these are both blends of unlike crudes. Results of tests on these oils mixed in various proportions are given in Table III, and it is seen that the correction factors are lower when the heavy oil is of naphthene than when it is of paraffin base.

TABLE III.—TESTS OF BLENDS OF UNLIKE CRUDES AT 55°C. (131°F.).

Oils	Per cent. of lighter oil in blend	Sp. gr. 15.6° / 15.6° C.	t	μ	$\frac{t}{t'}$	$\frac{\mu}{\mu'}$
$N + Q$	100	0.885	111.5	0.1965		
	90	.....	122.7	0.2202	0.945	0.940
	80	.....	134.0	0.2441	0.883	0.874
	70	.....	152.1	0.2817	0.853	0.845
	60	.....	181.1	0.3410	0.863	0.857
	50	.....	209.7	0.3990	0.846	0.842
	30	.....	290.2	0.565	0.839	0.837
	20	.....	343.6	0.674	0.847	0.837
	10	.....	424.0	0.838	0.873	0.877
	0	0.934	577.4	1.147	.....	.....
$P + M$	100	0.925	111.7	0.2060	.....	.....
	90	.....	126.6	0.2363	0.977	0.975
	85	.....	136.3	0.2557	0.972	0.971
	80	.....	144.2	0.2710	0.952	0.949
	70	.....	170.2	0.3220	0.958	0.957
	60	.....	203.8	0.3861	0.977	0.974
	50	.....	238.5	0.4520	0.971	0.968
	40	.....	278.9	0.530	0.960	0.962
	35	.....	299.7	0.568	0.950	0.951
	30	.....	323.2	0.612	0.943	0.945
	20	.....	377.1	0.710	0.930	0.931
	10	.....	445.6	0.836	0.936	0.936
	5	.....	481.1	0.901	0.925	0.923
	0	0.878	567.7	1.060	.....	.....
$N + M$	50	.....	219.5	0.4282	0.885	0.882
$P + Q$	50	.....	236.2	0.4376	0.962	0.959

DIAGRAMS FOR ESTIMATING CORRECTION FACTORS

Fig. 130 shows the results of tests. It will be noted that the time ratio, or correction factor, is not a minimum for the 50 per cent. blends which are indicated by vertical dotted lines. While Fig. 130 is convenient for interpolation in a given series of tests, it is not convenient for interpolation between one series and another.

In order to reduce the number of variables, it was decided to concentrate attention upon 50 per cent. blends. If the correction factor for this blend is known, the factors for other proportions may be estimated by sketching curves similar in shape to those of Fig. 130. Curves for the correction factor $\frac{\mu}{\mu'}$ come to a somewhat sharper point at the bottom. The variable due to nature of crude may be eliminated by using separate diagrams for naphthene and paraffin crudes, as the difference between them is not so great as to make the error in interpolating between them of serious amount.

Density may be eliminated by using poises instead of Saybolt viscosities. Then the only remaining variables are the viscosities of the two component oils, for which the correction factors for 50 per cent. blends are desired.

A blend of any proportion may be considered as a 50 per cent. blend of two other blends, as, for example, a 20 per cent. blend is a blend of two others, one containing 10 and the other 30 per cent. of the component in question. By estimating the viscosity of the 20 per cent. blend from the experimentally determined viscosities of the 10 and 30 per cent. blends, a new correction factor may be determined. The tests with gasoline (series

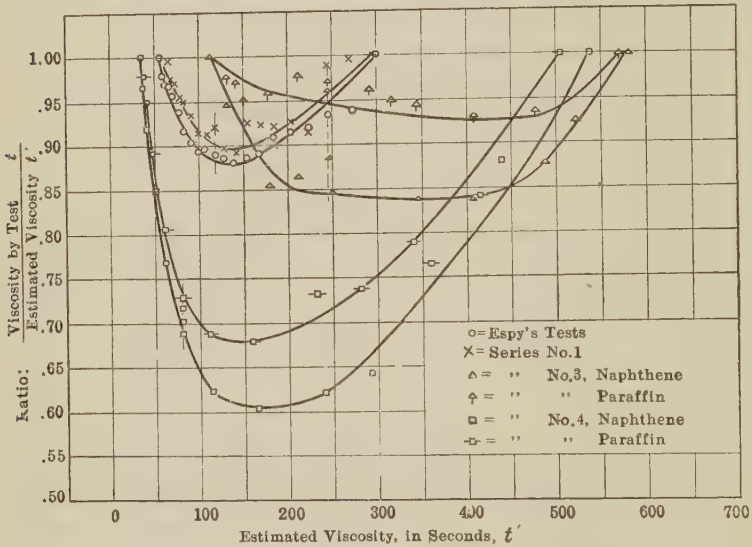


FIG. 130.

2), different crudes (series 3) and blends containing kerosene (series 4) were used in this manner to obtain correction factors for 50 per cent. blends, and the results are shown in Fig. 131.

The figure is divided into two sections by the diagonal line marked 1.00, the upper portion being for the blends $P + M$ and the lower for the blends $N + Q$. In plotting the diagram each point was marked with the corresponding value of $\frac{\mu}{\mu'}$ (not shown) and the lines drawn to conform as well as possible with these values, as in making a contour map. As might be expected from the preceding figure, the points for the upper portion of the diagram were more discordant than those for the lower.

The range of viscosities in Fig. 131 is greater than met with in actual blending operations. Excluding steam engine cylinder oils, or "cylinder stock," the viscosities vary from about 750 down to 80 seconds at 100°F. (37.8°C.), or, say, 1.5 to 0.12 poise. In the extreme case, therefore, the correction factor from Fig. 131 would be 0.75 for naphthene and 0.80 for paraffin crudes, corresponding to errors in viscosity of 33 and 25 per cent., respectively. The errors in Saybolt viscosity would be somewhat less.

The estimation of the viscosity of blends containing cylinder stock presents an additional difficulty on account of the error in measurement of viscosities due to the change of volume of the oil after leaving the outlet tube. Espy gives results of tests on two series of blends of oil of 300 and 55 seconds viscosity, the temperature of test being in one case 100°F. (37.8°C.) and in the other 210°F. (98.9°C.). Corresponding values in these series agreed within one second, and Espy apparently assumes that it would be entirely safe to use his diagram for any temperature. It is believed, however, that the possibility should be emphasized of serious disagreement between Fig. 131 and experimental results, when cylinder stock is used at temperatures approaching 100°C., unless precautions are taken to avoid the error above mentioned.

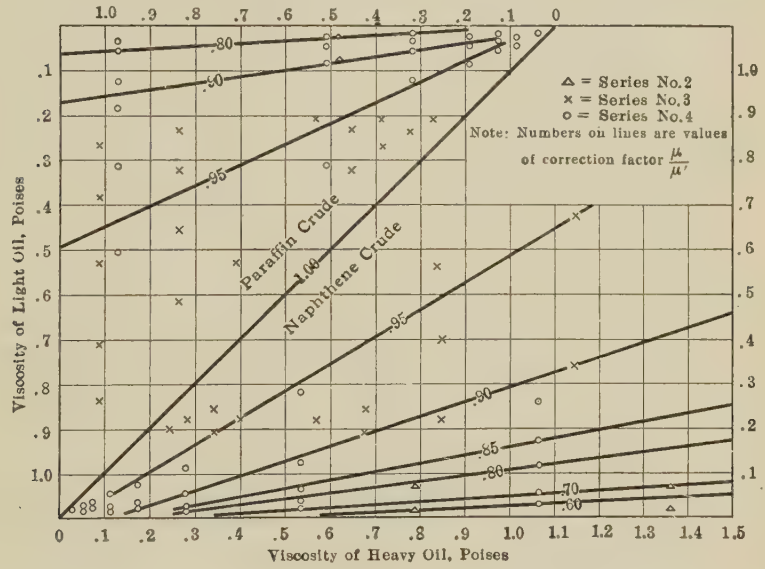


FIG. 131.

CONCLUSIONS

The evidence of the tests here recorded is that the rule that the logarithms of the viscosities in poises are additive may be made the basis of an estimate of the viscosity of a blend. The determination of viscosities in poises, from readings of the Saybolt viscosimeter, has been discussed in previous papers of the Bureau of Standards. The larger the difference in viscosity of the component oils, the greater will be the error in the logarithmic rule, the true viscosity being less than the estimated value. For equal differences of viscosity, the error increases as the viscosity of the lighter component decreases. The true viscosity is greater than that calculated on the assumption that fluidities are additive.

Since it was found that the error in the logarithmic rule depends upon the source of the component oils, it seemed preferable to use an uncorrected

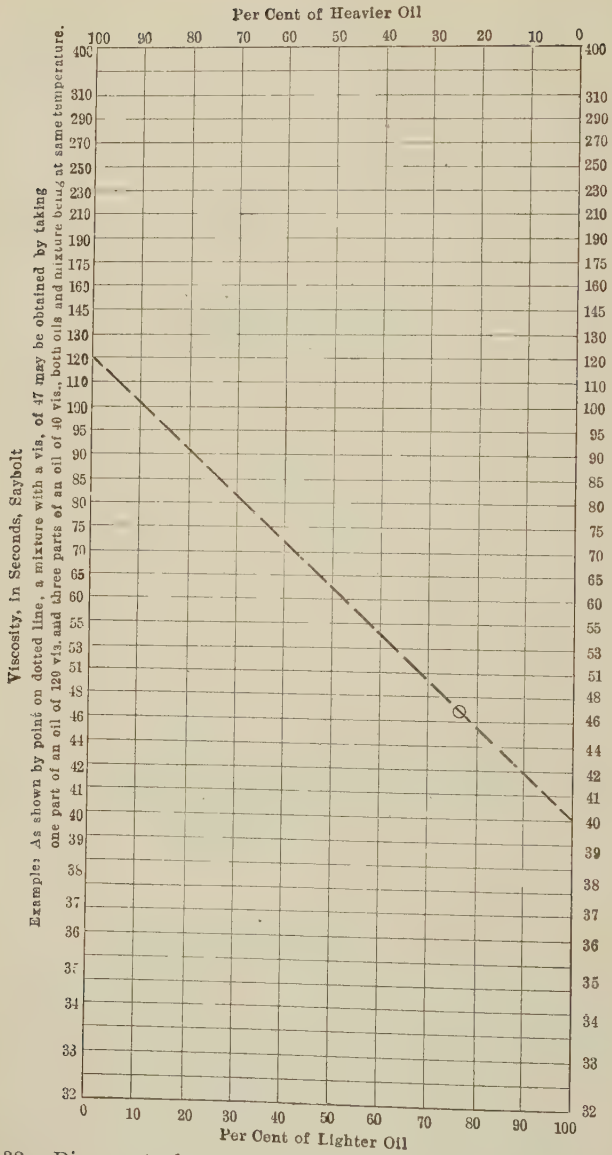

Per Cent of Heavier Oil
100 90 80 70 60 50 40 30 20 10 0
Viscosity, In Seconds, Saybolt
Example: As shown by point on dotted line, a mixture with a vis. of 47 may be obtained by taking one part of an oil of 120 vis. and three parts of an oil of 40 vis., both oils and mixture being at same temperature.
400 310 290 270 250 230 210 190 175 160 145 130 120 110 100 95 90 85 80 75 70 65 60 55 53 51 48 46 44 42 41 40 39 38 37 36 35 34 33 32
0 10 20 30 40 50 60 70 80 90 100
Per Cent of Lighter Oil

FIG. 132.—Diagram to determine the approximate viscosities of mixtures.

table or diagram calculated by this rule, and give data concerning variations in the correction factor, so that each user of the diagram could select the appropriate correction factor for the crudes at hand. Diagrams are given for finding the correction factor for 50 per cent. blends of oils of any crude or any viscosity, and for then estimating the correction factor for blends of other proportions. It is believed that by this means the error in estimating the viscosity of a blend will not greatly exceed the error in determining the viscosity of the component oils.

Specifications for Mechanism Lubricating Oil as Formulated by Committee XVII of the Signal Division of the American Railroad Association

SPECIFICATIONS FOR MECHANISM LUBRICATING OIL

1919

1. Material.—(*a*) The oil shall be guaranteed by the manufacturer to be straight run, refined and filtered from one hundred per cent. (100 per cent.) pure Pennsylvanian crude oil, obtained by fractional distillation, unmixed with any other substance, and not acid treated.

(*b*) The oil shall be not darker than four (4) (salmon) in color on the Saybolt colorimeter, neutral and free from vegetable or animal oils or fats, paraffin, alkali, or foreign matter.

(*c*) The oil shall conform to the following requirements:

1. Gravity. Baumé 32½° to 33½°.
2. Flash. Minimum 345°F. (174°C.).
3. Fire. Minimum 395°F. (202°C.).
4. Viscosity. 85 sec. minimum at 100°F. (38°C.); 30 sec. minimum at 212°F. (100°C.).
5. Cold. Cloud 10°F. (12°C.); Pour −5°F. (−15°C.); Flow −25°F. (−3.9°C.).
6. Moisture. None.
7. Evaporation. Not more than 2.75 per cent. at 212°F. (100°C.) for eight (8) hours.
8. Sulphur. None.
9. Acid. None.

2. Inspection.—Standard clauses submitted by the Committee on Standard Clauses and Sections are to be inserted, if approved by the Division.

3. Tests.—Standard clauses submitted by the Committee on Standard Clauses and Sections are to be inserted, if approved by the Division.

(*d*) Sample for test shall be taken from the bottom of the oil through an opening in the container or by the use of a thief.

(*e*) Tests shall be made in accordance with the following methods.

1. *Gravity Test.*—Gravity shall be made by the use of a hydrometer, Westphal balance or pycnometer. Observation shall be taken with the sample sixty (60) degrees F. (16°C.), compared with water at the same temperature. Correction for the buoyant effect of the atmosphere shall be made when necessary. (Degree Baumé may be obtained through the use of the formula adopted by the Bureau of Standards.)

2. *Flash Test.*—(*a*) The flash test shall be made in the Cleveland Open-Cup consisting of two parts, one the cup, the other a brass metal plate five (5) inches in diameter by one-fourth (¼) inch thick, slightly recessed in its upper face to receive the base of the cup. The metal plate and oil holder are usually mounted on a tripod to which a wire is attached for supporting the thermometer. The latter may be supported by a clamp on a ring stand or by a chain. The thermometer used shall be of the engraved chemical stem "bulb immersion" type, corrected for one (1) inch immersion and reading to approximately seven hundred (700) degrees F. (371°C.). The bulb shall be about three-eighths (⅜) inch long, not exceeding one-half (½) inch. The thermometer shall be suspended in the center of the cup, and free from the bottom by one-eighth (⅛) inch. Fill cup to within one-half (½) inch from the top. Avoid overheating locally by wiping the flange free of oil and not allowing oil to be spattered up the sides of the cup.

(*b*) The oil shall be heated at a uniform rate of ten (10) degrees F. per minute; and at approximately twenty-five (25) degrees F. below the expected flash, the test shall be applied at every rise of five (5) degrees F. by resting the gas tube on the edge of the cup and searching for the flash in a semicircle around the thermometer midway between it and the inside

of the cup, carrying the flame level with the top of the cup. The temperature at which flashes occur on the surface of the oil at two points or one that covers at least half of the surface and immediately goes out shall be recorded as the flash test.

(*c*) The testing flame is provided by using a slender tube tapered at one end to an opening approximately one-sixteenth (1/16) inch diameter. The flame should be in the form of a bead and not more than one-eighth (1/8) inch diameter.

3. *Fire Test.*—The fire test shall be made by continuing the flash test, the operation being the same except in the application of the test flame. The flame in the fire test shall be quickly brought to one-sixteenth (1/16) inch above the surface of the oil near the center of the cup and as quickly removed. The fire test is usually from forty (40) degrees F. (40°C.) to eighty (80) degrees F. (27°C.) higher than the flash test; the temperature to which the oil must be heated to cause it to give off gases which will burn continuously when a flame is applied above the surface of the oil, shall be the fire test.

4. *Viscosity Test*—The viscosity test shall be made by using the Saybolt Universal Viscosimeter. The oil tube shall be swabbed with the plunger accompanying the instrument by using oil from sample, making sure the overflow tube is not obstructed. Strain the oil into the oil tube at a temperature not above that of the bath, stir until the oil is of the test temperature throughout, remove the thermometer, remove the excess oil from the overflow chamber with the pipette and proceed with the test. The elapsed time in seconds required for the oil to fill the flask to the 60 c.c. mark on the neck shall be the viscosity of the oil.

5. *Cold Test.*—(*a*) The cold test shall be made by using a glass jar approximately one and one-fourth (1¼) inch in diameter and four (4) to five (5) inches high, provided with a tightly fitting cork. The glass jar shall be incased in a closely fitting metal jacket, which shall be provided at the bottom with a disc of cork or felt one-fourth (¼) inch thick.

(*b*) A bulb immersion type mercury thermometer having a bulb one-fourth (¼) inch to three-eighths (⅜) inch long shall be fitted securely in the cork so that the shaft will be held centrally in the jar with the tip of the bulb one-half (½) inch from the bottom.

(*c*) The oil shall be placed in the jar to a depth of about one and one-fourth (1¼) inch, or to a sufficient depth to reach about one-fourth (¼) inch above bulb of thermometer. Fit the cork tightly into the jar, put thermometer in cork, making sure that bottom of bulb is about one-half (½) inch from the bottom of the jar. Place jar in metal jacket and place jacket in freezing mixture.

(*d*) *Cloud.*—When near the expected cloud point, at every drop of two (2) degrees F. remove the jar from the jacket and inspect, being careful not to disturb the oil by removing the thermometer or otherwise. The cloud test of the oil shall be the temperature at which the lower half of the sample becomes opaque.

(*e*) *Pour.*—The test shall be continued and at each drop of temperature of (5) degrees F. remove the jar from jacket and tilt just enough to make it flow, and five (5) degrees higher than the temperature of the oil which has been cooled so that it will not flow when the jar is tipped to a horizontal position, shall be the pour test. The rate of cooling shall be such that the pour test is complete in about one-half (½) hour.

(*f*) *Flow.*—Continue lowering the temperature until the sample has frozen solid. The flow point shall be the lowest temperature at which the previously frozen oil will flow with a gradual rise in temperature.

6. *Moisture Test.*—The moisture test shall be made by taking a sample from the bottom of the container, which, when placed in a clean and dry glass or porcelain beaker and stirred without touching the sides or bottom with a one-fourth (¼) inch brass or iron rod heated to a cherry red, shall not cause a snapping sound.

7. *Evaporation Test.*—(*a*) The evaporation test shall be made by the use of Freas electrically heated oven, in which shall be placed near the outer edge of the revolving table ten (10) grams (15.4 grains avdp) of oil in an aluminum dish having the following dimensions:

Inside diameter—64 to 65 mm. (about 2.5 in.)

Inside height—12 to 14 mm. (about ½ in.)

Thickness of metal—1 to 1.5 mm. (about 0.050 in.)

Weight of dish—14 to 15 grams (about 216 to 232 grains avdp.)

(*b*) The evaporation shall be expressed in percentage of loss in weight to the initial weight of oil.

8. *Acid Test.*—(*a*) The acid test shall be made by agitating thoroughly a solution at the boiling-point of five (5) grams of the oil with twenty-five (25) c.c. of distilled water and twenty-five (25) c.c. of ninety-five (95) per cent. alcohol. A few drops of phenolphthalein shall be added as an indicator and the result, if colorless (indicating acid present), shall

be titrated with one-tenth (1/10) normal alkali, free from carbonate until permanently pink when agitated. The percentage of acid shall be calculated from reading the burette and shown in percentage of oleic acid or as an acid number (milligrams of potassium hydroxide required to saturate the free acid in one gram of oil).

One (1) c.c. of one-tenth (1/10) alkali (.0560 m/g of KOH = 0.0282 gram of oleic acid).

(*a*) (Alkali, one (1) c.c. of which is equivalent to 0.00993 gram of KOH and one (1) c.c. of which is equivalent to one-half (1/2) per cent. of oleic acid may be used).

(*b*) A strip of polished copper shall not tarnish when placed in a sample of oil at two hundred and twelve (212) degrees F. (100°C.) for eight (8) hours.

(*c*) A sample of oil shall not darken more than two (2) numbers on the Saybolt colorimeter when held for five (5) minutes at the flash-test heat.

4. Marking.—Standard clauses submitted by the Committee on Standard Clauses and Sections are to be inserted, if approved by the Division.

5. Packing.—The oil shall be put in clean, dry containers of the kind and capacity as specified on the purchaser's order sheet and shall be protected so as to permit convenient handling and to prevent loss or damage during shipment.

Specifications for Zero Fahrenheit Lubricating Oil as Formulated by Committee XVI of the Signal Division of the American Railroad Association

SPECIFICATIONS FOR ZERO FAHRENHEIT LUBRICATING OIL

1920

1. Purpose.—The purpose of this specification is to provide for a lubricating oil for signal mechanisms and crossing protection apparatus in territories where the temperature does not fall below zero (0) degrees Fahr. This oil shall not be used on relays or in dash oil pots. 1920.

2. Material.—(*a*) Oil shall be pale in color, neutral and free from vegetable or animal oils or fats, alkali or foreign matter. 1920.

(*b*) Oil shall conform to the following requirements:

1. Flash. Minimum 305°F. (151.7°C.).
2. Fire. Minimum 345°F. (173.9°C.).
3. Viscosity. 100 sec. minimum to 135 sec. maximum at 100°F. (37.8°C.) on the universal Saybolt viscosimeter.
4. Cold. Pour 0°F. (17.8°C.)
5. Moisture. None.
6. Sulphur. Free sulphur—None.
7. Acid. Not more than 0.03 of 1 per cent. calculated as SO_3. 1920.

3. Inspection.—(*a*) Purchaser may inspect the material at all stages of manufacture. 1919.

(*b*) Purchaser may inspect the completed product to determine that the requirements of this specification have been met. 1919.

(*c*) If the material has not been accepted at point of production, and if, upon arrival at destination, it does not meet the requirements of this specification, it may be rejected and the contractor, upon request, shall advise the purchaser what disposition is to be made of the defective material. The contractor shall pay all freight charges. 1919.

(*d*) If the purchaser is to make inspection at point of production it shall be so stated. 1917.

4. Tests.—(*a*) Tests may be made at point of production, or on samples submitted, and may also be made at destination. 1919.

(*b*) Contractor shall give the purchaser sufficient notice of time when material will be ready for testing. 1919.

(*c*) Contractor shall provide at point of production apparatus and labor for making the required tests under supervision of the purchaser. 1919.

(*d*) Sample for test shall be taken from the bottom of the oil through an opening in the container or by use of a thief. 1919.

(*e*) Tests shall be made in accordance with the following methods.

1. *Flash Test.*—(*a*) The flash test shall be made in the Cleveland open-cup, consisting of two parts, one the cup, the other a brass metal plate five (5) inches in diameter by one-fourth (1/4) inch thick, slightly recessed in its upper face to receive the base of the cup. The metal

plate and oil holder are usually mounted on a tripod to which a wire is attached for supporting the thermometer. The latter may be supported by a clamp on a ring stand or by a chain. The thermometer used shall be of the engraved chemical stem "bulb immersion" type, corrected for one (1) inch immersion and reading to approximately seven hundred (700) degrees F. (371°C.). The bulb shall be about three-eighths (3/8) inch long, not exceeding one-half (1/2) inch. The thermometer shall be suspended in the center of the cup, and free from the bottom by one-eighth (1/8) inch. Fill cup to within one-half (1/2) inch from the top. Avoid overheating locally by wiping the flange free of oil and not allowing oil to be spattered up the sides of the cup. 1920.

(*b*) The oil shall be heated at a uniform rate of ten (10) degrees F. per minute, and at approximately twenty-five (25) degrees F. below the expected flash the test shall be applied at every rise of five (5) degrees F. by resting the gas tube on the edge of the cup and searching for the flash in a semicircle around the thermometer, midway between it and the inside of the cup, carrying the flame level with the top of the cup. The temperature at which flashes occur on the surface of the oil at two points or one that covers at least half of the surface and immediately goes out shall be recorded as the flash test. 1920.

(*c*) The testing flame is provided by using a slender tube tapered at one end to an opening approximately one-sixteenth (1/16) inch diameter. The flame should be in the form of a bead and not more than one-eighth (1/8) inch diameter. 1920.

2. *Fire Test.*—The fire test shall be made by continuing the flash test, the operation being the same except in the application of the test flame. The flame in the fire test shall be quickly brought to one-sixteenth (1/16) inch above the surface of the oil near the center of the cup and as quickly removed. The fire test is usually from forty (40) degrees F. to eighty (80) degrees F. higher than the flash test, the temperature to which the oil must be heated to cause it to give off gases which will burn continuously when a flame is applied above the surface of the oil, shall be the fire test. 1920.

3. *Viscosity Test.*—The viscosity test shall be made by using the Saybolt universal viscosimeter. The oil tube shall be swabbed with the plunger accompanying the instrument by using oil from sample, making sure the outflow tube is not obstructed. Strain the oil into the oil tube at a temperature not above that of the bath, stir until the oil is of the test temperature throughout, remove the thermometer, remove excess oil from the overflow chamber with the pipette and proceed with the test. The elapsed time in seconds required for the oil to fill the flask to the 60 c.c.-mark on the neck shall be the viscosity of the oil. 1920.

4. *Cold Test.*—(*a*) The cold test shall be made by using a glass jar approximately one and one-fourth (1 1/4) inch in diameter and four (4) to five (5) inches high provided with a tightly fitting cork. The glass jar shall be incased in a closely fitting metal jacket which shall be provided at the bottom with a disk of cork or felt one-fourth (1/4) inch thick. 1920.

(*b*) A bulb immersion type mercury thermometer having a bulb one-fourth (1/4) inch to three-eighths (3/8) inch long shall be fitted securely in the cork so that the shaft will be held centrally in the jar with the tip of the bulb one-half (1/2) inch from the bottom. A toluol thermometer shall be used for temperatures lower than −20°F. (−20.9°C.).

(*c*-1) Material used in the freezing mixture varies with the temperature required to cause the oil to solidify. Use cracked ice for a temperature above 1.7°C. (35°F.). From −9.4°C. (15°F.) to −20.6°C. (−5°F.) use ice and salt in proportion of one to two. The salt shall be dry and fine enough to pass through a 20-mesh screen. For temperatures lower than −20.6°C. (−5°F.) use a mixture of solid carbon dioxide and acetone.

(*c*-2) The carbon dioxide-acetone mixture may be made as follows:

Place a sufficient amount of dry acetone in a covered copper or nickel beaker. Place the beaker in an ice-salt mixture and when the acetone reaches 10°F. or less, add solid carbon dioxide gently until the desired temperature is reached. To obtain the solid carbon dioxide, invert an ordinary liquefied carbon dioxide cylinder, open the valve carefully and let the gas flow into a chamois skin bag. Rapid evaporation will cause the carbon dioxide to solidify. 1920.

(*d*) The oil shall be placed in the jar, to a depth of about one and one-fourth (1 1/4) inch, or to a sufficient depth to reach about one-fourth (1/4) inch above bulb of thermometer. Fit the cork tightly into the jar, put the thermometer in cork, making sure that bottom of bulb is about one-half (1/2) inch from the bottom of the jar Place jar in metal jacket and place jacket in freezing mixture. 1920.

(*e*) *Pour.*—When near the expected pour point, at every drop in temperature of 5°F. remove the jar from the jacket and chill just enough to make it flow, and 5 per cent. higher

than the temperature of the oil which has been cooled, so that it will not flow when the jar is tipped to a horizontal position, shall be the pour test. 1920.

5. *Moisture Test.*—The moisture test shall be made by taking a sample from the bottom of the container, which, when placed in clean and dry glass or porcelain beaker and stirred without touching the sides or bottom with a one-fourth (¼) inch brass or iron rod heated to a cherry red, shall not cause a snapping sound. 1920.

6. *Acid Test.*—(*a*) The acid test shall be made by agitating thoroughly a solution at the boiling-point of five (5) grams of the oil with twenty-five (25) c.c. of distilled water and twenty-five(25)c.c. of ninety-five(95)per cent. neutralized alcohol. A few drops of phenolphthalein shall be added as an indicator and the result, if colorless (indicating acid present), shall be titrated with one-tenth (1/10) normal alkali, free from carbonate, until permanently pink when agitated. The percentage of acid shall be calculated from reading the burette and shown in percentage of SO_3 or as an acid number (milligrams of potassium hydroxide required to saturate the free acid in one gram of oil). One (1) c.c. of one-tenth (1/10) alkali equals four (4) m/g SO_3. 1920.

(*b*) A strip of polished copper shall not tarnish when placed in a sample of oil at two hundred and twelve (212) degrees F. (100°C.) for eight (8) hours. 1920.

5. Marking.—(*a*) Purchaser's order and requisition number, name of consignor and name and address of consignee shall be plainly marked on outside of package. 1916.

(*b*) Detail list of loose pieces, containers and their contents shall be furnished for each shipment. Where carload shipments are made, routing and car identification shall be shown. 1919.

(*c*) Where carload shipments are made, each package shall be marked with contents; order number and address may be omitted. 1919.

6. Packing.—The oil shall be put in clean dry containers of the kind and capacity as specified on the purchaser's order sheet and shall be protected, so as to permit convenient handling and to prevent loss or damage during shipment. 1920.

Specifications for 45° Below Zero Fahrenheit Lubricating Oil as Formulated by Committee XVI of the Signal Division of the American Railroad Association

SPECIFICATIONS FOR 45° BELOW ZERO FAHRENHEIT LUBRICATING OIL

1920

1. Purpose.—The purpose of this specification is to provide for a lubricating oil for signal mechanisms. This oil shall not be used in oil dash pots. 1920.

2. Material.—(*a*) The oil shall be pale in color, neutral and free from vegetable or animal oils or fats, alkali or foreign matter. 1920.

(*b*) The oil shall conform to the following requirements:

1. Flash. Minimum 290°F.(143.3°C.).
2. Fire. Minimum 325°F.(162.8°C.).
3. Viscosity. Minimum 80 sec. at 100°F. (37.8°C.) on the universal Saybolt viscosimeter.
4. Cold. Pour at −45°F.
5. Moisture. None.
6. Sulphur. Free sulphur—None.
7. Acid. Not more than 0.03 of 1 per cent. calculated as SO_3. 1920.

3. Inspection.—(*a*) Purchaser may inspect the material at all stages of manufacture. 1919.

(*b*) Purchaser may inspect the completed product to determine that the requirements of this specification have been met. 1919.

(*c*) If the material has not been accepted at point of production, and if, upon arrival at destination, it does not meet the requirements of this specification, it may be rejected and the contractor, upon request, shall advise the purchaser what disposition is to be made of the defective material. The contractor shall pay all freight charges. 1919.

(*d*) If the purchaser is to make inspection at point of production, it shall be so stated. 1917.

4. Tests.—(*a*) Tests may be made at point of production or on samples submitted, and may also be made at destination. 1919.

(*b*) Contractor shall give the purchaser sufficient notice of time when material will be ready for testing. 1919.

(*c*) Contractor shall provide, at a point of production, apparatus and labor for making the required tests under supervision of the purchaser. 1919.

(*d*) Sample for test shall be taken from the bottom of the oil through an opening in the container by use of a thief. 1919.

(*e*) Tests shall be made in accordance with the following methods.

1. *Flash Test.*—(*a*) The flash test shall be made in the Cleveland open-cup, consisting of two parts, one the cup, the other a brass metal plate five (5) inches in diameter by one-fourth (¼) inch thick, slightly recessed in its upper face to receive the base of the cup. The metal plate and oil holder are usually mounted on a tripod to which a wire is attached for supporting the thermometer. The latter may be supported by a clamp on a ring stand or by a chain. The thermometer used shall be of the engraved chemical stem "bulb immersion" type, corrected for one (1) inch immersion and reading to approximately seven hundred (700) degrees F. (371°C.). The bulb shall be about three-eighths (⅜) inch long, not exceeding one-half (½) inch. The thermometer shall be suspended in the center of the cup, and free from the bottom by one-eighth (⅛) inch Fill the cup to within one-half (½) inch from the top. Avoid overheating locally by wiping the flange free of oil and not allowing oil to be spattered up the sides of the cup. 1920.

(*b*) The oil shall be heated at a uniform rate of ten (10) degrees F. per minute, and at approximately twenty-five (25) degrees F. below the expected flash the test shall be applied at every rise of five (5) degrees F. by resting the gas tube on the edge of the cup and searching for the flash in a semicircle around the thermometer, midway between it and the inside of the cup, carrying the flame level with the top of the cup. The temperature at which flashes occur on the surface of the oil at two points or one that covers at least half of the surface and immediately goes out shall be recorded as the flash test. 1920.

(*c*) The testing flame is provided by using a slender tube tapered at one end to an opening approximately one-sixteenth (1/16) inch in diameter. The flame should be in the form of a bead and not more than one-eighth (⅛) inch diameter. 1920.

2. *Fire Test.*—The fire test shall be made by continuing the flash test, the eopration being the same except in the application of the test flame. The flame in the fire test shall be quickly brought to one-sixteenth (1/16) inch above the surface of the oil near the center of the cup and as quickly removed. The fire test is usually from forty (40) degrees F. (44°C.) to eighty (80) degrees F. (27°C.) higher than the flash test, the temperature to which the oil must be heated to cause it to give off gasses which will burn continuously when a flame is applied above the surface of the oil, shall be the fire test. 1920.

3. *Viscosity Test*—The viscosity test shall be made by using the Saybolt universal viscosimeter. The oil tube shall be swabbed with the plunger accompanying the instrument by using oil from samples, making sure the outflow tube is not obstructed. Strain the oil into the oil tube at a temperature not above that of the bath, stir until the oil is of the test temperature throughout, remove the thermometer, remove the excess oil from the overflow chamber with the pipette and proceed with the test. The elapsed time in seconds required for the oil to fill the flask to the 60 c.c. mark on the neck shall be the viscosity of the oil. 1920.

4. *Cold Test.*—(*a*) The cold test shall be made by using a glass jar approximately one and one-fourth (1¼) inch in diameter and four (4) to five (5) inches high provided with a tightly fitting cork. The glass jar shall be incased in a closely fitting metal jacket which shall be provided at the bottom with a disk of cork or felt one-fourth (¼) inch thick. 1920.

(*b*) A bulb immersion type mercury thermometer having a bulb one-fourth (¼) inch to three-eighths (⅜) inch long shall be fitted securely in the cork so that the shaft will be held centrally in the jar with the tip of the bulb one-half (½) inch from the bottom. A toluol thermometer shall be used for temperatures lower than −20°F. 1920.

(*c*-1) Materials used in the freezing mixture vary with the temperature required to cause the oil to solidify. Use cracked ice for a temperature above 1.7°C. (35°F.); from −9.4°C. (15°F.) to 20.6°C. (−5°F.) use ice and salt in proportion of one to two. The salt should be dry and fine enough to pass through a twenty (20) mesh screen. For temperature lower than −20.6°C. (−5°F.) use a mixture of solid carbon dioxide and acetone. 1920.

(*c*-2) The carbon dioxide-acetone mixture may be made as follows: Place a sufficient amount of dry acetone in a covered copper or nickel beaker. Place the beaker in an ice-salt mixture and when the acetone reaches 10°F. or less, add solid carbon dioxide gently until the desired temperature is reached. To obtain the solid carbon dioxide, invert an ordinary

liquefied carbon dioxide cylinder, open the valve carefully and let the gas flow into a chamois skin bag. Rapid evaporation will cause the carbon dioxide to solidify. 1920.

(*d*) The oil shall be placed in the jar to a depth of about one and one-fourth (1¼) inch, or to a sufficient depth to reach about one-fourth (¼) inch above the bulb of thermometer. Fit the cork tightly into the jar, put thermometer in cork, making sure that bottom of bulb is about one-half (½) inch from the bottom of the jar. Place jar in metal jacket and place jacket in freezing mixture. 1920.

(*e*) *Pour.* When near the expected pour point, at every drop in temperature of 5°F. remove the jar from the jacket and chill just enough to make it flow, and 5 per cent. higher than the temperature of the oil which has been cooled so that it will not flow when the jar is tipped to a horizontal position shall be the pour test. 1920.

5. *Moisture Test.*—The moisture test shall be made by taking a sample from the bottom of the container, which, when placed in a clean and dry glass or porcelain beaker and stirred without touching the sides or bottom with a one-fourth (¼) inch brass or iron rod heated to a cherry red, shall not cause a snapping sound. 1920.

6. *Acid Test.*—(*a*) The acid test shall be made by agitating thoroughly a solution at the boiling-point of five (5) grams of the oil with twenty-five (25) c.c. of distilled water and twenty-five(25) c.c. of ninety-five(95) per cent. neutralized alcohol. A few drops of phenolphthalein shall be added as an indicator and the result, if colorless (indicating acid present), shall be titrated with one-tenth (1/10) normal alkali, free from carbonate, until permanently pink when agitated. The percentage of acid shall be calculated from reading the burette and shown in percentage of SO_3 or as an acid number (milligrams of potassium hydroxide required to saturate the free acid in one gram of oil). One (1) c.c. of one-tenth (1/10) alkali equals four (4) m/g SO_3. 1920.

(*b*) A strip of polished copper shall not tarnish when placed in a sample of oil at two hundred and twelve (212) degrees F. (100°C.) for eight (8) hours. 1920.

5. Marking.—(*a*) Purchaser's order and requisition number, name of consignor and name and address of consignee shall be plainly marked on outside of package. 1916.

(*b*) Detail list of loose pieces, containers and their contents, shall be furnished for each shipment. Where carload shipments are made, routing and car identification shall be shown. 1919.

(*c*) Where carload shipments are made, each package shall be marked with contents; order number and address may be omitted. 1919.

6. Packing.—The oil shall be put in clean dry containers of the kind and capacity as specified on the purchaser's order sheet and shall be protected so as to permit convenient handling and to prevent loss or damage during shipment. 1920.

General Requirements for Lubricants. (Data from Lockhart's "American Lubricants.")

		Flash	Fire	Cold test	Say-bolt time	Other requirements
		Cleveland cup				
Miscellaneous combustion engines.	Small stationary gas engines.	400°F.			220–270 @ 100°F.	Viscous neutrals. Should not turn black when heated 15 minutes at flash-point.
	Large stationary gas engines.	400°F.			250–450 @ 100°F.	Viscous neutrals. Should not turn black when heated 15 minutes at flash-point.
	R. R. section cars....				350 or above at 100°F.	
	Motorcycle engines...				350–800 @ 100°F.	
	Aeroplane engines....	above 400°F.		not above 15°F.	400–550 @ 100°F.	Minimum amount of matter insoluble in petroleum ether after heating at 250°C. for 2½ hrs.
Automobile engines.....	Light automobile cylinder oils.	380°F. 415°F.			162–215 @ 100°F.	Should not turn black when heated for 15 minutes at the flash-point.
	Medium automobile cylinder oils.	385°F. 430°F.			196–285 @ 100°F.	Should not turn black when heated for 15 minutes at the flash-point.
	Heavy automobile cylinder oils.	420°F. 430°F.			262–435 @ 100°F.	Should not turn black when heated for 15 minutes at the flash-point.
General automobile lubrication.	Transmission.....	550°F. 600°F.	600°F. 675°F.	30°F.	160–220 @ 210°F.	For heavy cars and trucks a semifluid grease is used.
	Differential.....					Semifluid transmission grease.
	Worm drives.....					Heavy gear grease of cylinder-stock base.
	Roller bearings.....					Medium or soft grade of fiber or cup grease. For heavy trucks a heavy fiber or sponge grease.

Electrical machinery	Small dynamos and motors, 5–35 h.p.	above 380°F.		below 20°F.	140–180 @ 100°F.	Viscous neutrals.
	Large dynamos and motors above 50 h.p.	400°F.			160–220 @ 100°F.	Viscous neutrals.
	Transformer oil	over 340°F.		below 20°F.	80–140 @ 100°F.	Non-viscous neutral. Should have small evaporation loss. Should be free from water, acid, and gumming tendency.
	Very small dynamos and motors.				90–110 @ 100°F.	Non-viscous neutrals.
Steam engines	Cylinder oils	485°F. 585°F.			100–220 @ 210°F.	Residues (cylinder stock) mixed with high as 12 per cent. fatty oil. The stock or finished oil should not show precipitation with gasoline.
	General lubrication	400°F.			180–250	For forced feed should be non-emulsifying engine oil.
Marine engines	Cylinders				125–140 @ 210°F.	Low fatty oil content. For superheated cylinders little if any fatty oil added to stock. Viscosity, 180–250 @ 210°F.
	General lubrication				above 350 @ 100°F.	Heavy engine oil compounded with 20–30 per cent. blown rape seed oil.
Steam railways	Locomotive journals					Very heavy journal grease.
	General engine lubrication.				220–270 @ 100°F.	Engine oils.
	Car oils	375°F. 400°F.			60–95 @ 210°F.	Cheap grade of oil residue, called well oil, black oil, reduced oil. Used for journals, etc. Summer car oils, vis. 80–95 @ 210°F.; winter car oils, vis. 60–65 @ 210°F.

		Flash (Cleveland cup)	Fire (Cleveland cup)	Cold test	Say-bolt time	Other requirements
Textile mills	Spindle oils, light	275°F. 285°F.	320°F. 325°F.		46–53 @ 100°F.	Neutral oils.
	Spindle oils, heavy	345°F. 355°F.	390°F. 395°F.		93–104 @ 100°F.	Neutral oils.
	Regular spindle oil	325°F.	380°F.		64 @ 100°F.	Neutral oils.
	Loom oils	320°F. 415°F.	425°F. 490°F.		100–225 @ 100°F.	Neutral and engine oils. Neatsfoot, lard or olive oil are often used alone or mixed with mineral oils for lace machines.
	Shafting oils				220–290 @ 100°F.	Very heavy engine oil.
	Turbine lubrication	400°F.			125–160 @ 100°F.	Neutral oil the best, low cold test, should not emulsify, contain no sulphur, and should not blacken when heated 15 minutes at flash-point.
	Knitting mills					For small parts oil of 100 viscosity or below @ 100°F. For heavier needle plates, a neutral oil of 140–180 viscosity @ 100°F.
Miscellaneous	Air compressors	above 400°F.			160–200 200–250 @ 100°F.	Should have minimum carbonization when heated several hours at the flash-point. Low cold test.
	Ammonia compressors	at least 375°F.		0°F.	50– 90 @ 100°F.	Low cold test.
	Cutting tools					Kerosene or paraffin oils compounded with high as 20 per cent. cotton-seed or lard oil. Also "soluble" oils emulsified in water.

Forms for Reporting the Results of Tests on Bituminous Road Materials

FILE CARDS

UNITED STATES DEPARTMENT OF AGRICULTURE,
OFFICE OF PUBLIC ROADS AND RURAL ENGINEERING,
Washington, D. C.

Sample No......

Report on.....
Identification mark.....
Known as.....
Submitted by.....
Examined for.....

		% by vol.	% by wt.	Character.
General character.....	Distillation—			
Specific gravity 25°/25°C.....	Water.....	.....	.....	.....
Melting point °C.....	First light oils...(–110°C.)	.....	.....	.....
Float test at.... °C.....; at.... °C.....	Second light oils (110–170°C.)	.....	.....	.....
Viscosity Engler at.... °C..... c.c. specific..	Heavy oils.....(170–270°C.)	.....	.....	.....
Bitumen soluble in CS_2.....	Heavy oils.....(270–300°C.)	.....	.....	.....
Free carbon (insoluble in CS_2).....	Pitch residue.....	.....	.....	.....
Ash.....				
.....	Total.....	.....	.....	.....

.....
.....
Remarks.....
.....
.....
Date received....., Date reported.....

Analyst.....

UNITED STATES DEPARTMENT OF AGRICULTURE,
OFFICE OF PUBLIC ROADS AND RURAL ENGINEERING,
Washington, D. C.

Sample No......

Report on.....
Identification mark.....
Known as.....
Submitted by.....

General character.....	Loss at....; °C.....hours.....
Specific gravity 25°/25°C.....	Character of residue.....
Melting point °C.....	Consistency of residue { Float test at.... °C.... / Penetration at 25°C. (mm.)
Flash point °C.....	
Burning point °C.....	
Viscosity at.... °C.....; c.c. specific.....	Bitumen soluble in CS_2.....
Ductility at 25°C. (cm.).....	Organic matter insoluble.....
Float test at... °C. time...; at... °C. time...	Inorganic matter insoluble.....
Penetration at 25°C. (mm.).....	% of total bitumen insol. in 86° naphtha.....
.....	% of total bitumen insol. in CCl_4.....
.....	% fixed carbon.....

Remarks.....
.....
Date received....., Date reported.....

Analyst.....

UNITED STATES DEPARTMENT OF AGRICULTURE,
OFFICE OF PUBLIC ROADS AND RURAL ENGINEERING,
Washington, D. C.

Sample No......

Report on.....
Identification mark.....
Known as.....
Submitted by.....
Examined for.....

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.....
Remarks.....
.....
.....
Date received....., Date reported.....

Analyst.....

REPORT BLANKS

UNITED STATES DEPARTMENT OF AGRICULTURE,
OFFICE OF PUBLIC ROADS AND RURAL ENGINEERING,
Washington, D. C.

Date.....

Sample No......
Report on.....
Identification mark.....
Known as.....
Submitted by.....
Examined for.....
.....
.....
.....
.....
.....
.....
Remarks.....

UNITED STATES DEPARTMENT OF AGRICULTURE,
OFFICE OF PUBLIC ROADS AND RURAL ENGINEERING,
Washington, D. C.

Date.....

Sample No......
Report on.....
Identification mark.....
Known as.....
Submitted by.....
General characteristics.....
Specific gravity 25°/25°C.....
Melting point °C.....
Flash point °C.....
Burning point °C.....
Viscosity Engler at.... °C.... c.c. specific.....
Consistency { Float test.... °C., time....; .... C°., time.....
Consistency { Penetration 25°C., 100 gms., 5 seconds.....
Loss at 163°C., 5 hours.....
Character of residue.....
Consistency of residue { Float test.... °C. time....; .... °C., time.....
Consistency of residue { Penetration 25°C., 100 gms., 5 seconds.....
Bitumen soluble in CS_2 (total bitumen).....
Organic matter insoluble.....
Inorganic matter insoluble.....
Per cent. of total bitumen insoluble in 86°B. naphtha.....
Per cent. of total bitumen insoluble in CCl_4.....
Per cent. fixed carbon.....
Distillation —

	Character	% by vol.	% by wt.
Water.....	.....	.....	.....
First light oils...(–110°C.).....	.....	.....	.....
Second light oils (110°–170°C.).....	.....	.....	.....
Heavy oils.....(170°–270°C.).....	.....	.....	.....
Heavy oils.....(270°–300°C.).....	.....	.....	.....
Pitch.....	.....	.....	.....

Remarks.....

Standard Test for Penetration of Bituminous Materials, Adopted in 1911, Revised in 1916, by the American Society for Testing Materials

STANDARD TEST FOR PENETRATION OF BITUMINOUS MATERIALS

I. DEFINITION

1. Penetration.—Penetration is defined as the consistency of a bituminous material, expressed as the distance that a standard needle vertically penetrates a sample of the material under known conditions of loading, time and temperature. Where the conditions of test are not specifically mentioned, the load, time and temperature are understood to be 100 g., 5 seconds, 25°C. (77°F.), respectively, and the units of penetration to indicate hundredths of a centimeter.

II. APPARATUS

2. Container.—The container for holding the material to be tested shall be a flat-bottom, cylindrical dish, 55 mm. ($2\frac{3}{16}$ in.) in diameter and 35 mm. ($1\frac{3}{8}$ in.) deep.[1]

3. Needle.—The needle[2] for this test shall be of cylindrical steel rod 50.8 mm. (2 in.) long and having a diameter of 1.016 mm. (0.04 in.) and turned on one end to a sharp point having a taper of 6.35 mm. ($\frac{1}{4}$ in.).

4. Water Bath.—The water bath shall be maintained at a temperature not varying more than 0.1°C. from 25°C. (77°F.). The volume of water shall be not less than 10 liters and the sample shall be immersed to a depth of not less than 10 cm. (4 in.) and shall be supported on a perforated shelf not less than 5 cm. (2 in.) from the bottom of the bath.

5. Apparatus for Penetration.—Any apparatus which will allow the needle to penetrate without appreciable friction, and which is accurately calibrated to yield results in accordance with the definition of penetration, will be acceptable.

6. Transfer Dish for Container.—The transfer dish for container shall be a small dish or tray of such capacity as will insure complete immersion of the container during the test. It shall be provided with some means which will insure a firm bearing and prevent rocking of the container.

III. PREPARATION OF SAMPLE

7. Preparation of Sample.—The sample shall be completely melted at the lowest possible temperature and stirred thoroughly until it is homogeneous and free from air bubbles. It shall then be poured into the sample container to a depth of not less than 15 mm. ($\frac{5}{8}$ in.). The sample shall be protected from dust and allowed to cool in an atmosphere not lower than 18°C. (65°F.) for one hour. It shall then be placed in the water bath along with the transfer dish and allowed to remain one hour.

[1] This requirement is fulfilled by the American Can Company's Gill style ointment box, deep pattern, 3 oz. capacity.

[2] *Journal of Agricultural Research*,. **5**, 1125.

IV. TESTING

8. Testing.—(*a*) In making the test the sample shall be placed in the transfer dish filled with water from the water bath of sufficient depth to completely cover the container. The transfer dish containing the sample shall then be placed upon the stand of the penetration machine. The needle, loaded with specified weight, shall be adjusted to make contact with the surface of the sample. This may be accomplished by making contact of the actual needle point with its image reflected by the surface of the sample from a properly placed source of light. Either the reading of the dial shall then be noted or the needle brought to zero. The needle is then released for the specified period of time, after which the penetration machine is adjusted to measure the distance penetrated.

At least three tests shall be made at points on the surface of the sample not less than 1 cm. (⅜ in.) from the side of the container and not less than 1 cm. (⅜ in.) apart. After each test the sample and transfer dish shall be returned to the water bath and the needle shall be carefully wiped toward its point with a clean, dry cloth to remove all adhering bitumen. The reported penetration shall be the average of at least three tests whose values shall not differ more than four points between maximum and minimum.

(*b*) When desirable to vary the temperature, time and weight, and, in order to provide for a uniform method of reporting results when variations are made, the samples shall be melted and cooled in air as above directed. They shall then be immersed in water or brine, as the case may require, for one hour at the temperature desired. The following combinations are suggested:

At 0°C. (32°F.), 200-g. weight, 60 seconds.
At 46.1°C. (115°F.), 50-g. weight, 5 seconds.

Tentative Method for Determination of Softening-Point of Bituminous Materials Other Than Tar Products, Issued 1916 by the American Society for Testing Materials

TENTATIVE METHOD FOR DETERMINATION OF SOFTENING-POINT OF BITUMINOUS MATERIALS OTHER THAN TAR PRODUCTS

1. Definition.—The Ring-and-Ball method shall be used.

2. Apparatus.—The apparatus shall consist of a brass ring 15.875 mm. (⅝ in.) in diameter, 6.35 mm. (¼ in.) deep, 2.38125 mm. (3/32 in.) wall, suspended 25.40 mm. (1 in.) above bottom of beaker; a steel ball 9.525 mm. (⅜ in.) in diameter, weighing between 3.45 and 3.50 g.; a standardized thermometer; a glass beaker, approximately 600-c.c. capacity.

3. Method.—Carefully melt the sample and fill the ring with the material to be tested. Remove any excess. Place the ball in the center of the ring and suspend in the beaker containing approximately 400 c.c. of water at a temperature of 5°C. (41°F.). Arrange the thermometer bulb within ½ in. of the sample and at the same level. Apply heat *uniformly* over bottom of the beaker in quantity sufficient to raise the temperature 5°C.

(9°F.) per minute. Record the temperature at starting the test and every minute thereafter until the test is completed. The rate of heating is very important. The softening point is the temperature at which the specimen has dropped 1 in. Successive tests should average within 3°C. For temperatures above 90°C., glycerin shall be used instead of water.

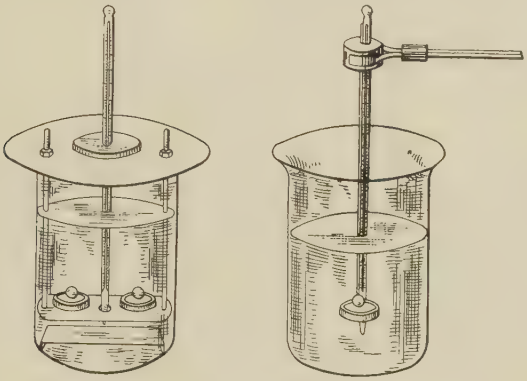

FIG. 133.—Apparatus for the determination of softening-point.

Standard Test for Loss on Heating of Oil and Asphaltic Compounds, Adopted in 1911, Revised in 1916, by the American Society for Testing Materials

STANDARD TEST FOR LOSS ON HEATING OF OIL AND ASPHALTIC COMPOUNDS

The amount lost by oils and asphaltic compounds when they are heated in an oven at a temperature of 163°C. (325°F.), ±1°C., shall be determined by heating 50 g. of the water-free substance contained in a flat-bottom dish, the inside dimensions of which are approximately 55 mm. ($2\frac{3}{16}$ in.) in diameter, by 35 mm. (about $1\frac{3}{8}$ in.) deep (3-oz. Gill style ointment box, deep pattern), for 5 hours.

The oven in which the substance is to be heated shall be brought to the prescribed temperature before the sample is introduced, and the temperature of the sample under test shall be regarded as that of a similar quantity of the same material immediately adjoining it in the oven, in which the bulb of a standardized thermometer is immersed.

The oven may be either of circular or rectangular form and the source of heat either gas or electricity.

The samples under test shall rest in the same relative position in a single row upon a perforated circular shelf, 24.8 cm. (9.75 in.) in diameter, as shown in Fig. 134, suspended by a vertical shaft midway in the oven, which is revolved by mechanical means at the rate of from 5 to 6 revolutions per minute.

NOTE

If additional periods of heating are desired, it is recommended that they be made in successive increments of 5 hours each.

If the residue after heating is to be tested for penetration, the sample should be thoroughly mixed by stirring until it is cool, and thereafter manipulated in accordance with the directions of the Standard Test for Penetration of Bituminous Materials (Serial Designation: D 5) of the American Society for Testing Materials (see page 385).

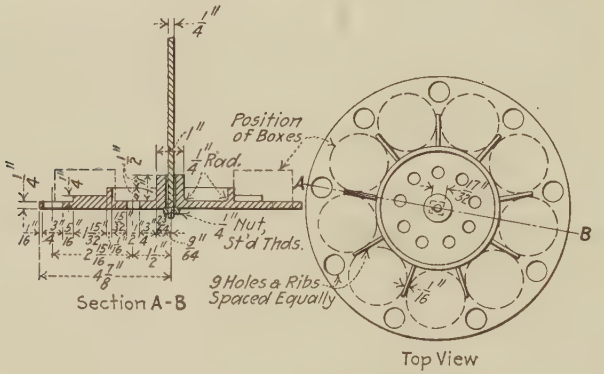

FIG. 134.—Aluminum shelf.

Standard Method for Distillation of Bituminous Materials Suitable for Road Treatment, Adopted in 1916, Revised in 1918, by the American Society for Testing Materials

STANDARD METHOD FOR DISTILLATION OF BITUMINOUS MATERIALS SUITABLE FOR ROAD TREATMENT

1. Sampling.—The sample as received shall be thoroughly stirred and agitated, warming, if necessary, to insure a complete mixture before the portion for analysis is removed.

2. Dehydration.—If the presence of water is suspected or known, the material shall be dehydrated before distillation. About 500 c.c. of the material are placed in an 800-c.c. copper still provided with a distilling head connected with a water-cooled condenser. A ring burner is used, starting with a small flame at the top of the still, and gradually lowering it, if necessary, until all the water has been driven off. The distillate is collected in a 200-c.c. separatory funnel with the tube cut off close to the stopcock. When all the water has been driven over and the distillate has settled out, the water is drawn off and the oils returned to the residue in the still. The contents of the still shall have cooled to below 100°C. before the oils are returned, and they shall be well stirred and mixed with the residue.

3. Apparatus.—The apparatus shall consist of the following standard parts:

(*a*) *Flask.*—The distillation flask shall be a 250-c.c. Engler distilling flask, having the following dimensions:

Diameter of bulb	8.0 cm.
Length of neck	15.0 cm.
Diameter of neck	1.7 cm.
Surface of material to lower side of tubulature	11.0 cm.
Length of tubulature	15.0 cm.
Diameter of tubulature	0.9 cm.
Angle of tubulature	75 deg.

A variation of 3 per cent. from the above meaurements will be allowed.

(*b*) *Thermometer.*—The thermometer shall conform to the following requirements:

It shall be made of thermometric glass of a quality equivalent to suitable grades of Jena or Corning make. It shall be thoroughly annealed. It shall be filled above the mercury with inert gas which will not act chemically on or contaminate the mercury. The pressure of the gas shall be sufficient to prevent separation of the mercury column at all temperatures of the scale. There shall be a reservoir above the final graduation large enough so that the pressure will not become excessive at the highest temperature. The thermometer shall be finished at the top with a small glass ring or button suitable for attaching a tag. Each thermometer shall have for identification the maker's name, a serial number, and the letters "A.S.T.M. Distillation."

The thermometer shall be graduated from 0 to 400°C. at intervals of 1°C. Every fifth graduation shall be longer than the intermediate ones, and every tenth graduation beginning at zero shall be numbered. The graduation marks and numbers shall be clear-cut and distinct.

The thermometer shall conform to the following dimensions:

Total length, maximum	385 mm.
Diameter of stem	7 mm.; permissible variation, 0.5 mm.
Diameter of bulb, minimum	5 mm.; and shall not exceed diameter of stem.
Length of bulb	12.5 mm.; permissible variation, 2.5 mm.
Distance from 0° to bottom of bulb	30 mm.; permissible variation, 2.5 mm.
Distance from 0° to 400°	295 mm.; permissible variation, 10 mm.

The accuracy of the thermometer when delivered to the purchaser shall be such that when tested at full immersion the maximum error from 0 to 200°C. shall not exceed the following:

From 0 to 200°C	0.5°C.
From 200 to 300°C	1.0°C.
From 300 to 375°C	1.5°C.

The sensitiveness of the thermometer shall be such that when cooled to a temperature of 74°C. below the boiling-point of water at the barometric pressure, at the time of test, and plunged into free flow of steam, the meniscus shall pass the point 10°C. below the boiling-point of water in not more than 6 seconds.

The thermometer shall be set up as for the distillation test, using water, naphthalene and benzophenone as distilling liquids. The correctness of the thermometer shall be checked at 0 and 100°C. after each third distillation until seasoned.

(*c*) *Condenser.*—The condenser tube shall have the following dimensions:

Adapter	70 mm.
Length of straight tube	185 mm.
Width of tube	12–15 mm.
Width of adapter end of tube	20–25 mm.

(*d*) *Stands.*—Two iron stands shall be provided, one with a universal clamp for holding the condenser, and one with a light grip arm with a cork-lined clamp for holding the flask.

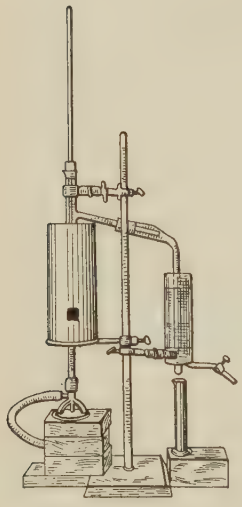

Fig. 135. — Apparatus for the determination of distillation.

(*e*) *Burner and Shield.*—A bunsen burner shall be provided, with a tin shield 20 cm. long by 9 cm. in diameter. The shield shall have a small hole for observing the flame.

(*f*) *Cylinders.*—The cylinders used in collecting the distillate shall have a capacity of 25 c.c., and shall be graduated in 0.1 c.c.

4. Setting Up the Apparatus.—The apparatus shall be set up as shown in Fig. 135, the thermometer being placed so that the top of the bulb is opposite the middle of the tubulature. All connections should be tight.

5. Method.—One hundred cubic centimeters of the dehydrated material to be tested shall be placed in a tared flask and weighed. After adjusting the thermometer, shield, condenser, etc., the distillation is commenced, the rate being so regulated that 1 c.c. passes over every minute. The receiver is changed as the mercury column just passes the fractionating point. The following fractions should be reported:

Start of distillation to 110°C.
110 to 170°C.
170 to 235°C.
235 to 270°C.
270 to 300°C.
Residue.

To determine the amount of residue, the flask is weighed again when distillation is complete. During the distillation the condenser tube shall be warmed when necessary to prevent the deposition of any sublimate. The percentages of fractions should be reported both by weight and by volume.

Provisional Method for the Determination of Soluble Bitumen, Adopted August 21, 1911, by the American Society for Testing Materials

PROVISIONAL METHOD FOR THE DETERMINATION OF SOLUBLE BITUMEN[1]

Drying the Sample and Preparing it for Analysis.—It was decided, owing to the great variety of conditions met with in bituminous compounds, that it is impossible to specify any one method of drying that would be satisfactory in every case. It is therefore supposed that the material for analysis has been previously dried either in the laboratory or in the process of refining or manufacture, and that water, if present, exists only as moisture in the hygroscopic form.

The material to be analyzed, if hard and brittle, is ground and spread in a thin layer in a suitable dish (iron or nickel will answer every purpose) and kept at a temperature of 125°C. for one hour. In the case of paving mixtures and road materials, where it is not desirable to crush the rock or sand grains, a lump may be placed in the drying oven until it is thoroughly heated through, when it can be crushed down into a thin layer and dried as above. If the material under examination contains any hydrocarbons at all volatile at this temperature, it will of course be necessary to resort to other means of drying. Tar or oils may be dehydrated by distillation and the water-free distillate returned to the residue and thoroughly incorporated with it.

Analysis of Sample.—After drying, from 2 to 15 grams (as may be necessary to insure the presence of 1 to 2 grams of pure bitumen) are weighed into a 150-c.c. tared Erlenmeyer flask, and treated with 100 c.c. of carbon disulphide. The flask is then loosely corked and shaken from time to time until all large particles of the material have been broken up. It is then set aside for 48 hours to settle. The solution is decanted into a similar flask that has been previously weighed. As much of the solvent is poured off as possible without disturbing the residue. The contents of the first flask are again treated with fresh carbon disulphide, shaken as before, and then put away with the second flask for 48 hours to settle.

The liquid in the second flask is then carefully decanted upon a weighed Gooch curcible, 3.2 cm. in diameter at the bottom, fitted with an asbestos filter, and the contents of the first flask are similarly treated. The asbestos filter is made of ignited long-fiber amphibole, packed in the bottom of a Gooch crucible to the depth of not over ⅛ in. In filtering no vacuum is to be used and the temperature is to be kept between 20° and 25°C. After passing the liquid contents of both flasks through the filter, the residue on the filter is thoroughly washed and the residues remaining in them are shaken with more fresh carbon disulphide and allowed to settle for 24 hours, or until it is seen that a good subsidation has taken place. The solvent in both flasks is then again decanted through the filter and the

[1] The Committee, in presenting the Provisional Method for the Determination of Soluble Bitumen, wish it understood that they do not recommend it as the best for general use, as it is longer and in many cases gives no better results than other more expeditious methods, but only as a method to be resorted to in case of dispute, as it seems to have the widest range of applicability of any of the methods considered. Moreover, they wish it to be understood that with some classes of materials the method will show a lower percentage of soluble bitumen than many of the shorter methods.

residues remaining in them are washed until the washings are practically colorless. All washings are to be passed through the Gooch crucible.

The crucible and both flasks are then dried at 125°C. and weighed. The filtrate containing the bitumen is evaporated, the bituminous residue burned, and the weight of the ash thus obtained added to that of the residue in the two flasks and the crucible. The sum of these weights deducted from the weight of substance taken gives the weight of soluble bitumen.

Specifications for Asphalt

Those given by the Association for Standardizing Paving Specifications in January, 1912, are as follows:

Refined Asphalt.—The refined asphalt to be used for paving mixtures herein required shall be derived as follows:

1. By heating, if requiring refinement, crude, natural, solid asphalt, to a temperature of not over 450°F., until all the water has been driven off. Crude, natural, solid asphalt shall be construed to mean any natural mineral bitumen, either pure or mixed with foreign matter, from which through natural causes in the process of time the light oils have been driven off until it has a consistency harder than 100 penetration at 77°F. At least 98.5 per cent. of the contained bitumen in the refined asphalt which is soluble in cold carbon disulphide shall be soluble in cold carbon tetrachloride. In no case shall such asphalt be prepared at the refinery with any product not hereinafter provided for.

2. By the careful distillation of asphaltic petroleum with continuous agitation until the resulting bitumen has a consistency not harder than 30 penetration at 77°F.

(*a*) All shipments of material shall be marked with a lot number and penetration, and ten samples taken at random from each lot shall not vary more than 15 per cent. from the average penetration, providing no part of any shipment shall be below 30 penetration at 77°F.

(*b*) The solid bitumen so obtained shall be soluble in CCl_4 to the extent of 98.5 per cent.

(*c*) When 20 grams of the material are heated for five hours at a temperature of 325°F. in a tin box 2.5 in. in diameter, after the manner officially prescribed, it shall lose not over 5 per cent. by weight nor shall the penetration at 77°F. after such heating be less than one-half the original penetration.

(*d*) The solid bitumen at a penetration of 50 shall have a ductility of not less than 20 cm. nor more than 85 cm. at 77°F. If the penetration varies from 50 an increase of at least 2 cm. in ductility will be required for each 5 points in penetration above 50, and a corresponding allowance will be made below 50 penetration. This test shall be made with a briquette of cross-section of 1 sq. cm., the material being elongated at the rate of 5 cm. per minute. (Dow moulds.)

NOTE.—Combinations of asphaltic bitumens having the ductility and other characteristics above mentioned are admitted under Section 2.

3. Refined asphalt produced by combining crude natural asphalt with either of the following:

(*a*) Residuums produced by the distillation of petroleum oils as specified under fluxes.

(*b*) Asphalts produced by the distillation of petroleum oils as specified.

In the use of these mixtures of refined asphalts for asphaltic cement, only asphaltic or semi-asphaltic fluxes shall be used, except in those cases where the solid natural asphalt is of such character when mixed with paraffin flux without the addition of any other material, it will produce an asphaltic cement complying with the requirements set forth under that head. In such cases any of the fluxes elsewhere specified may be used.

The preparation and refining of all asphalt admitted under these specifications shall be subject to such inspection at the paving plant and refineries as the proper city officials shall direct.

The fluxing material may be a paraffin, a semi-asphaltic or an asphaltic residue which shall be tested with and found suitable for the asphalts to be used.

The residuums shall have a penetration greater than 350°F. with a No. 2 needle at 77°F. under 50 grams weight for one second.

All residuums shall be soluble in cold CCl_4 to the extent of 99 per cent. and must remain soft after heating for 5 hours at 400°F.

(*a*) The paraffin residue shall have a specific gravity of 0.92 to 0.94 at 77°F. It shall not flash below 350°F. when tested in the N. Y. State Closed Oil Tester, and shall not volatilize more than 5 per cent. when heated for 5 hours at 325°F. in a tin box 2.5 in. in diameter, as officially prescribed. The residue after heating shall flow at 77°F. and shall be homogeneous and shall show no coarse crystals.

(*b*) Semi-asphaltic residuum shall have the same general characteristics as the paraffin residuum, except that it shall have a specific gravity of 0.94 to 0.98 at 77°F.

(*c*) Asphaltic residuum shall have the same general characteristics as the paraffin residuum, except that the specific gravity shall not be less than 0.98 nor more than 1.04 at 77°F. The asphaltic residuum after evaporation at 500°F. to a solid of 50 penetration shall have a ductility of not less than 30 cm. (Dow method.)

Asphaltic Cement

The asphaltic cement prepared from the materials above designated shall be made up from the refined asphalts and the flux, where flux must be used in such proportions as to produce an asphaltic cement of suitable degree of penetration. The proportion of the refined asphalt comprising the cement shall in no case be less than 40 per cent. by weight.

When the weight of flux in the asphaltic cement prepared from solid natural asphalt exceeds 25 per cent. thereof, asphaltic or semi-asphaltic flux shall be used.

Refined asphalts and flux comprising the asphaltic cement shall, when required, be weighed separately in the presence of the authorized inspectors or agents.

Refined asphalts and flux used in preparing the cement shall be melted together in a kettle ranging from 250° to not over 375°F. and shall be thoroughly agitated when hot by air, steam or mechanical appliances, until the resulting cement has become thoroughly mixed into a homogeneous mass. The agitation must be continued during the entire period of preparing the mixtures. Cement shall always be of uniform consistency, and if any portion should settle in the kettle between intervals of using the same, it must be thoroughly agitated before being drawn for use.

(*a*) The asphaltic cement shall have a penetration of 40 to 75, which shall be varied within these limits to adapt it to the particular asphalts used in the paving mixture and to the traffic and other conditions.

(*b*) When 20 grams of the asphaltic cement of the penetration to be used in the paving mixture shall be heated for 5 hours at a temperature of 325°F., in an oven as officially prescribed, there must not be volatilized more than 5 per cent. of the bitumen present, nor shall the penetration at 77°F. after such heating be less than one-half of the original penetration.

(*c*) A briquette of the asphalt cement when at a penetration of 50 having cross-sections of 1 sq. cm. shall elongate to the extent of not less than 20 cm. nor more than 85 cm. at 77°F. If the asphaltic cement as used in the paving mixture varies from 50 penetration, an increase of, at least 2 cm. in ductility will be required for each 5 points in penetration above 50, and a corresponding allowance will be made below 50 penetration. (Dow moulds.)

Tentative Specifications for Asphalt, Primer and Coal-Tar Pitch for Use in Damp-Proofing and Waterproofing, Issued 1917 by the American Society for Testing Materials

TENTATIVE SPECIFICATIONS FOR ASPHALT FOR USE IN DAMP-PROOFING AND WATERPROOFING

1. Material Covered.—These specifications cover asphalt for use in damp-proofing and waterproofing, designated as type A, recommended for use under uniformly moderate temperature conditions.

I. PROPERTIES

2. Melting-Point.—The melting-point shall be between 38 and 60°C. (100 and 140°F.) as determined by the ring-and-ball method in water bath, and shall be specified for one of the following classes: 54.5 to 60°C. (130–140°F.); 46 to 54.5°C. (115–130°F.); 38 to 46°C. (100–115°F.). The Tentative Method for Determination of Softening-Point of Bituminous Materials other than Tar Products (D 36–16 T) of the American Society for Testing Materials shall be used (see page 386).

3. Penetration.—The penetration at 25°C. (77°F.) under load of 100 g. for 5 seconds, as determined by the Standard Test for Penetration of Bituminous Materials (D 5)[1] of the American Society for Testing Materials, shall be not less than 50 nor more than 125.

The penetration shall bear the following relation to the melting-point:

Penetration of 50 to 75 for melting-points between 54.5 and 60°C. (130 and 140°F.).

Penetration of 75 to 100 for melting-points between 46 and 54.5°C. (115 and 130°F.).

Penetration of 100 to 125 for melting-points between 38 and 46°C. (100 and 115°F.).

4. Ductility.—The ductility[2] at 25°C. (77°F.), when a briquette of the material having a minimum cross-section of 1 sq. cm. is pulled apart at the rate of 5 cm. per minute, shall not be less than 30 cm.

5. Specific Gravity.—The specific gravity shall not be more than 1.08 at 25/25° C. (77/77°F.).

6. Soluble in Carbon Disulphide.—The bitumen soluble in cold carbon disulphide, as determined by the method in Section 10, shall not be less than 95 per cent.

7. Loss on Heating.—The loss of a 50-g. sample on heating at 163°C. (325°F.) for 5 hours, as determined by the Standard Test for Loss on Heating of Oil and Asphaltic Compounds (D 6)[3] of the American Society for Testing Materials, shall not exceed 1 per cent. The penetration of the residue from this test shall be not less than 50 per cent. of the original penetration specified in Section 3.

8. Ash.—The ash, as determined by the method in Section 10, shall not exceed 4 per cent.

II. METHODS OF TESTING

9. Specific Gravity.—The specific gravity may be determined by any approved method but some method similar to the following is recommended:

Weigh accurately in air a small platinum pan or crucible supported in a wire sling and suspended by a fine platinum wire or silk thread; record the weight as weight *A*. Then weigh it in a like manner completely immersed in freshly distilled water at 25°C. (77°F.), except for the fine platinum wire or silk thread. Record this weight as weight *B*. Then bring the crucible to red heat, cool and almost fill with asphalt which has been melted at the lowest possible temperature. Cool the crucible and contents to room temperature, place in the sling and weigh in air. Record the weight as weight *C*. Then weigh crucible and contents at 25°C. (77°F.) suspended as before in water, and record this weight as weight *D*. From these four weights the specific gravity may be calculated from the following formula:

$$\text{Specific Gravity} = \frac{C - A}{(C - A) - (D - B)}.$$

10. Bitumen Soluble in Cold Carbon Disulphide; Ash.—For this test weigh accurately 1 to 2 g. of asphalt into an Erlenmeyer flask. Pour 100 c.c. of c. p. carbon disulphide into the flask in small portions, with continual agitation, until all lumps disappear and nothing adheres to the bottom. Cork the flask and set aside for 15 minutes. Then filter the solution by suction through an asbestos pad in a Gooch crucible (the best size of crucible for this test is 4.4 cm. wide at the top tapering to 3.6 cm. at the bottom, and 2.5 cm. deep), which has been previously prepared and weighed. Care shall be exercised in decanting the liquid portion. Stop the decantation at the first sign of sediment coming out. Wash the sides of the flask with a small amount of fresh carbon disulphide and bring the sediment upon the felt, using a "policeman," if necessary, to remove all adhering material. Then wash the material on the mat with carbon disulphide until the washings are colorless and continue suction until the odor of carbon disulphide is hardly detectable. Then clean the outside of the crucible with a cloth moistened with a small amount of solvent. Dry the crucible with contents for one-half hour at 105°C. (221°F.), cool in a desiccator and weigh.

The total weight of insoluble material includes both organic and inorganic insoluble If organic insoluble material is present, heat the crucible to red heat until no carbonaceous particles remain—leaving only the mineral ash—cool and weigh. The percentage soluble in carbon disulphide, the organic insoluble, if there is any present, and the ash should total 100 per cent. Each should be reported. See page 385.

[1] **1918** Book of A. S. T. M. Standards. See page 385.

[2] A detailed description of the mold and method to be followed is given in Appendix IV, Report of Committee D-4 on Road Materials, *Proc. Am. Soc. Test. Mat.*, **15**, i, 349 (1915).

[3] **1918** Book of A. S. T. M. Standards. See page 387.

TENTATIVE SPECIFICATIONS FOR PRIMER FOR USE WITH ASPHALT FOR USE IN DAMP-PROOFING AND WATERPROOFING

1. Material Covered.—These specifications cover primer for use, when specified, with asphalt for use in damp-proofing and waterproofing.

2. Primer.—The primer shall consist of an asphaltic base, complying in every respect with the Tentative Specifications for Asphalt for Use in Damp-proofing and Waterproofing (Type A) for use below grade, which shall be thinned to ordinary paint consistency with a petroleum distillate having an end-point on distillation not above 260°C. (500°F.). Not more than 20 per cent. of this petroleum distillate shall distil under 120°C. (248°F.).

The distillation shall be carried out according to the Standard Tests for Paint Thinners other than Turpentine (D 28) of the American Society for Testing Materials (see page 343).

TENTATIVE SPECIFICATIONS FOR COAL-TAR PITCH FOR USE IN DAMP-PROOFING AND WATERPROOFING

1. Material Covered.—These specifications cover coal-tar pitch for use in damp-proofing and waterproofing, designated as type A, recommended for use under uniformly moderate temperature conditions.

I. PROPERTIES

2. Melting-Point.—The melting-point, as determined by the cube method in water bath, Section 9, shall be between 49 and 60°C. (120 and 140°F.). In specifying the melting-point desired within the above limits, a variation of not more than 2.5°C. (5°F.) in either direction will be permitted.

3. Penetration.—The penetration at 25°C. (77°F.) under load of 100 g. for 5 seconds, as determined by the Standard Test for Penetration of Bituminous Materials (D 6)[1] of the American Society for Testing Materials, shall not be less than 20 nor more than 120.

4. Ductility.—The ductility[2] at 25°C. (77°F.) when a briquette of the material having a minimum section of 1 sq. cm. is pulled apart at the rate of of 5 cm. per minute, shall not be less than 40 cm.

5. Loss on Heating.—The loss of a 20-g. sample on heating at 163°C. (325°F.) for 5 hours, as determined by the Standard Test for Loss on Heating of Oil and Asphaltic Compounds (D 6)[3] of the American Society for Testing Materials, on pitch of melting-point between 49 and 54.5°C. (120 and 130°F.), shall not exceed 9 per cent.; and on pitch of melting-point between 54.5 and 60°C. (130 and 140°F.), shall not exceed 7 per cent.

6. Specific Gravity.—The specific gravity of the pitch at 25/25°C. (77/77°F.) shall not exceed the limits 1.24 and 1.34.

The specific gravity at 60/60°C. (140/140°F.) of the distillate to 355°C. (671°F.) shall not be less than 1.06.

7. Soluble Matter.—The matter soluble in hot toluol-benzol, as determined by the method in Section 12, shall not be less than 65 nor more than 85 per cent.

8. Ash.—The ash, as determined by the method in Section 13, shall not exceed 1 per cent.

II. METHODS OF TESTING

9. Melting-Point.—The melting-point shall be the temperature at which the pitch formed in a ½-in. cube and suspended in a vessel of water 1 in. above the bottom shall touch the bottom of this vessel. The cube is formed by pressing the pitch in a suitable mold. Remove the cube from the mold and suspend on the lower arm of a No. 12 B. & S. gage wire bent at right angles. The wire should be passed through the center of two opposite faces of the cube. Immerse the cube in 400 c.c. of freshly boiled distilled water at a temperature of 15.5°C. (60°F.) contained in a 600-c.c. beaker. Maintain this temperature until the pitch is the same temperature as the water. Apply heat in such a manner that the temperature of the water is raised 5°C. (9°F.) per minute, until the pitch touches the bottom of the beaker. At this point the temperature, recorded by a thermometer whose bulb shall be level with the cube, shall be considered as the melting-point of the pitch.

[1] **1918** Book of A. S. T. M. Standards. See page 385.

[2] A detailed description of the mold and method to be followed is given in Appendix IV, Report of Committee D-4 on Road Materials, *Proc. Am. Soc. Test. Mat.*, **15**, i, 349 (1915).

[3] **1918** Book of A. S. T. M. Standards; use a 20-g. sample instead of the 50-g. sample specified. See page 387.

10. Specific Gravity.—The specific gravity may be determined by any approved method but some method similar to the following is recommended:

Weigh accurately in air a small platinum pan or crucible supported in a wire sling and suspended by a fine platinum wire or silk thread; record the weight as weight *A*. Then weigh it in a like manner completely immersed in freshly distilled water at 25°C. (77°F.), except for the fine platinum wire or silk thread. Record this weight as weight *B*. Then bring the crucible to red heat, cool and almost fill with pitch which has been melted at the lowest possible temperature. Cool the crucible and contents to room temperature, place in the sling and weigh in air. Record the weight as weight *C*. Then weigh crucible and contents at 25°C. (77°F.) suspended as before in water, and record this weight as weight *D*. From these four weights the specific gravity may be calculated from the following formula:

$$\text{Specific Gravity} = \frac{C-A}{(C-A)-(D-B)}.$$

11. Specific Gravity of Distillate.—Distil in a glass or metal retort, preferably of 8-oz. capacity, at least 100 g. of the pitch to 355°C. (671°F.) vapor temperature, catching the distillate. The specific gravity may be determined by any approved method, but the following is recommended:

Clean a 10-c.c. specific-gravity bottle; dry and weigh. Fill it with distilled water and heat to 60°C. (140°F.) as described below; dry and weigh. Then fill the bottle with distillate, immerse in water and heat slowly to 60°C. (140°F.), maintaining this temperature for at least 5 minutes. Dry, cool and weigh. From these weights the specific gravity may be determined from the following formula:

$$\text{Specific Gravity} = \frac{\text{Weight of bottle} + \text{distillate} - \text{weight of bottle}}{\text{Weight of bottle} + \text{water} - \text{weight of bottle}}.$$

12. Matter Insoluble in Hot Toluol-Benzol Extraction.[1]—In testing materials of 5 per cent. or more insoluble matter 5 g. should be taken for the test. With lesser percentages 10 g. should be used. Weigh out the amount in a 100-c.c. beaker, and digest with about 50 c.c. of c. p. toluol on the steam bath, for a period not to exceed 30 minutes. If the solution is kept hot and constantly stirred, the digestion can be completed very rapidly. Weigh a filter cup, previously prepared, in a weighing bottle and place in a carbon filter-tube over a beaker or flask. Decant the toluol-tar mixture through the thimble and wash with hot c. p. toluol until clean, using some form of "policeman" which is unaffected by toluol, for the purpose of detaching any residue which may adhere to the beaker. Finally, wash the cup with hot c. p. benzol and then, after draining, cover with a cap of filter paper or alundum, and place in the extraction apparatus in which the c. p. benzol is used as solvent. Continue the extraction until the descending benzol is colorless. Remove the thimble, take off the cap, dry in the steam oven and weigh in the weighing bottle after cooling in the desiccator. The balance used for this work should be accurate to at least ½ mg. It is well to examine the insoluble residue for foreign matter such as wood, slivers, pieces of bagging, etc. If such foreign matter is present, the test should be rejected.

13. Ash.—This determination is made by burning to ash a 1-g. sample of the material in a weighed platinum crucible or dish of sufficient size. Apply heat gently until the pitch ignites, then withdraw the heat. After the pitch ceases to burn, apply the heat again until the residue is burned free of carbon. Then cool the crucible and contents, weigh, and determine the ash.

Specifications for Sheet Asphalt Paving, Adopted October 12, 1916, by the American Society of Municipal Improvements

GENERAL DESCRIPTION

1. Upon the foundation prepared and laid as elsewhere herein specified, shall be laid the pavement proper. This shall consist of:

1. A binder course .... inches in thickness when compressed.
2. An asphalt wearing surface .... inches in thickness when compressed.

MATERIALS

2. The materials used must comply with the requirements of these specifications and be suitable for use upon the street or streets to be paved. They shall be mixed in definite

[1] A full description of a simple apparatus for this test may be found in *J. Ind. Eng. Chem.*, **6**, 279.

proportions by weight, depending upon their character and the traffic upon the street, and such materials and proportions must be satisfactory to the engineer.

3. Methods of Testing.—All tests herein specified must be conducted according to official methods on file in the office of the engineer. All penetrations at 77°F. are expressed in hundredths of a centimeter and are to be taken (except where otherwise specified) with a number two needle acting for five (5) seconds without appreciable friction under a total weight of one hundred (100) grams.

4. Refined Asphalts.—The refined asphalts admitted under these specifications shall be prepared from a natural mineral bitumen, either solid or liquid, or from combinations thereof, by such methods of refining as will produce a product complying with the requirements hereinafter given.

The preparation and refining of all asphalts admitted under these specifications shall be subject to such inspection at the paving plants and refineries as the engineer may direct. Every refined asphalt admitted under these specifications, if required by the engineer, shall be equal in quality to the recognized standard for its particular kind or type of asphalt. If desired, the contractor may use an asphalt cement prepared at the refinery. To be acceptable this asphalt cement must comply with the foregoing general requirements for refined asphalt, as well as requirements (*a*), (*b*), (*c*), (*d*), and (*e*) for asphalt cement.

Asphalt obtained by the refining of natural liquid bitumens shall not be reduced in the refining process to a penetration at 77°F. of less than 30.

All refined asphalts admitted under these specifications must comply with the following requirements:

(*a*) All shipments of refined asphalt of any one kind shall have the batch number plainly marked on each package or container and shall be uniform in consistency and composition and shall not vary from maximum to minimum more than fifteen (15) points in penetration at 77°F.

(*b*) Ninety-eight and one-half (98½) per cent. of the total bitumen of all refined asphalts shall be soluble in carbon tetrachloride.

(*c*) When made into an asphalt cement by the use of such materials and methods as are described in these specifications, they must produce an asphalt cement complying with all the requirements elsewhere set forth herein for asphalt cements.

5. Fluxes.—These shall be the residues obtained by the distillation of paraffin asphaltic or semi-asphaltic petroleums. They shall be of such character that they will combine with the asphalt to be used to form an acceptable and approved asphalt cement complying with the requirements of these specifications. All residuums must pass the following general tests:

(*a*) They must have a penetration greater than three hundred and fifty (350) with a No. 2 needle at 77°F. under fifty (50) grams weight for one second.

(*b*) They shall have a specific gravity at 77°F. between 0.92 and 1.02.

(*c*) When twenty (20) grams of the flux are heated for five (5) hours at 325°F. in a tin box two and one-quarter (2¼) inches in diameter and three-quarters (¾) of an inch deep after the manner officially prescribed, the loss shall not exceed five (5) per cent. by weight and the residue left after such heating shall flow at 77°F.

(*d*) They shall not flash below 350°F. when tested in a closed oil tester.

(*e*) They shall be soluble in carbon tetrachloride to the extent of not less than ninety-nine (99) per cent.

6. Binder Stone.—This shall be clean, hard, broken stone, free from any particles that have been weathered, or are soft. If the stone does not contain the proper amount of material passing the one-half (½) inch screen, the deficiency may be made up by the addition of gravel or sand. Ninety-five (95) per cent. of the binder aggregate shall pass a screen having circular openings whose diameter shall be three-quarters (¾) the thickness of the binder course to be laid. The remaining five (5) per cent. shall not exceed in their largest dimension the thickness of the binder course to be laid. The binder aggregate shall be so graded from coarse to fine as to have the following mesh composition (sieves to be used in the order named):

Passing:			
10-mesh	15 to 35 per cent.		
½-in. circular opening and retained on 10-mesh	20 to 50 per cent.	Total passing ½ in.	35 to 85 per cent.

The above limits as to mesh composition are intended to provide for such permissible variations as may be rendered necessary by the available sources of supply and the character of the work to be done. The mesh composition and character of the stone may be varied, within the limits above specified, at the discretion of the engineer, depending upon the kind of asphalt used and the traffic conditions upon the street or streets to be paved.

7. Sand.—The sand shall be hard, clean grained and moderately sharp. On sifting it shall have the following mesh composition (sieves to be used in the order named):

Passing:		
200 mesh	0 to 5 per cent.	Total passing 80-mesh and retained on 200-mesh 20 to 40 per cent.
100 mesh and retained on 200 mesh	10 to 25 per cent.	
80 mesh and retained on 100 mesh	6 to 20 per cent.	
50 mesh and retained on 80 mesh	5 to 40 per cent.	
40 mesh and retained on 50 mesh	5 to 30 per cent.	
30 mesh and retained on 40 mesh	5 to 25 per cent.	Total passing 10-mesh and retained on 40-mesh 12 to 45 per cent.
20 mesh and retained on 30 mesh	5 to 15 per cent.	
10 mesh and retained on 20 mesh	2 to 10 per cent.	
8 mesh and retained on 10 mesh	0 to 5 per cent.	

On very light traffic streets a coarser sand may be used with the approval of the engineer, but in no case shall a sand be employed that contains less than a total of fifteen (15) per cent. passing an 80-mesh sieve, such total to contain not more than five (5) per cent. (calculated on the original sand) passing a 200-mesh sieve, or a mixture of seventy-five (75) per cent. of sand of the character above specified and twenty-five (25) per cent. of stone screenings passing a one-quarter (¼)-inch screen and retained on a 10-mesh screen, may be employed.

The above limits as to mesh composition are intended to provide for such permissible variations as may be rendered necessary by the available sources of supply and the character of the work to be done, it being understood that the coarser permissible grading is intended for use on light and medium traffic streets only, and that for heavy traffic streets the finer grading shall be required. The mesh composition and character of the sand shall be varied, within the limits above specified by the engineer, depending upon the kind of asphalt used and the traffic conditions upon the street or streets to be paved.

Filler.—This shall be thoroughly dry limestone dust or portland cement, the whole of which shall pass a 30 mesh per linear inch screen and at least 66 per cent. of which shall pass 200 mesh per linear inch screen. The surface mixture shall contain from 6 to 20 per cent. of this filler, depending upon the kind of sand and asphalt used and the traffic conditions upon the street or streets to be paved.

8. Samples.—One (1) pound samples of the refined asphalt, petroleum flux and asphalt cement that the contractor proposes to use in his work, together with a statement as to the source, character and proportions of the materials composing them, must be handed in with his bid and no contract shall be awarded to any bidder whose samples do not comply in every respect with these specifications. No asphalt other than that specified in his bid shall be used by any contractor except with the written consent of the engineer and provided that it complies in all respects with the requirements of these specifications.

In addition to the samples submitted with the bid, other samples taken from and actually representative of the refined asphalt, petroleum flux, sand filler and binder stone to be used upon the street shall be submitted to the engineer before the use of such materials in the work is permitted. Except at his option, no work on binder or surface shall be commenced within three weeks from the date when such samples were submitted and in no case shall they be used until they have been examined and approved by him. Whenever, during the course of the work, new deliveries of paving materials are received by the contractor, samples of these shall at once be submitted to the engineer and their use in the work will not be permitted until they have been examined and approved by him.

ASPHALT CEMENT

9. Preparation.—The asphalt cement shall be composed of refined asphalt, or asphalts and flux, where flux is required, of the character elsewhere herein specified and must be of a suitable degree of penetration.

The proper proportions of the refined asphalt, or asphalts, and flux, shall be melted together at a temperature between 275 and 400°F. and thoroughly agitated by suitable

appliances until they are completely blended into a homogeneous asphalt cement. Thereafter, the asphalt cement must not be heated to a temperature exceeding 350°F. If the asphalt cement contains material that will separate by subsidence while it is in a molten condition, it must be thoroughly agitated before drawing from storage and while in use in the supply kettles. Excessive agitation with steam or air which will injure the cement must not be used.

The refined asphalt or asphalts and flux comprising the asphalt cement shall, when required, be weighed separately in the presence of the authorized inspectors or agents of the engineer:

10. Requirements.—The asphaltic cement shall comply with the following requirements:

(*a*) It shall be thoroughly homogeneous.

(*b*) It shall have a penetration at 77°F. of from 30 to 55 for heavy traffic streets and 55 to 85 for light traffic streets, depending upon the sand and asphalt used and the local climate conditions.

(*c*) It shall not flash below 350°F. when tested in a closed oil tester.

(*d*) When twenty (20) grams of the asphalt cement are heated for five (5) hours at 325°F. in a tin box two and one-quarter (2¼) inches in diameter and three-quarters (¾) of an inch deep, after the manner officially prescribed, the loss shall not exceed five (5) per cent. by weight and the penetration at 77°F. of the residue left after such heating must not be less than one-half the penetration at 77°F. of the original sample before heating.

(*e*) Either the asphalt cement or its pure bitumen when made into a briquette (Dow mold) shall, at 50 penetration (77°F.), have a ductility of not less than 30 cm. at 77°F.; the two ends of the briquette to be pulled apart at the uniform rate of 5 cm. per minute.

When the asphalt cement as used has a penetration other than 50 at 77°F., an increased ductility of 2 cm. will be required for every five points in penetration above 50 penetration and a corresponding allowance will be made below 50 penetration.

BINDER

11. Preparation.—The binder shall be composed of stone, or stone and sand, and asphalt cement of the character elsewhere herein specified and mixed in proper proportions. The stone, or stone and sand, and the asphalt cement shall be heated separately to such a temperature as will give, after mixing, a binder of the proper temperature for the materials employed. The stone when used must be at a temperature between 225 and 350°F. The asphalt cement and stone shall be thoroughly mixed by machinery until a homogeneous mixture is produced in which all the articles are thoroughly coated with asphalt cement.

12. Laying.—The binder mixture prepared in the manner above described shall be brought to the work in wagons covered with canvas or other suitable material and upon reaching the street shall have a temperature between 200°F. and 325°F. The temperature of the binder mixture within these limits shall be regulated according to the temperature of the atmosphere and the working of the binder. On reaching the street it shall at once be dumped on the concrete and then be deposited roughly in place by means of hot shovels, after which it shall be uniformly spread by means of hot iron rakes and then at once be thoroughly compacted by tamping or rolling. Tamping shall only be permitted in those places which are inaccessible to a roller. Rolling shall be carried on continuously at the rate of not more than three hundred (300) square yards per hour per roller until a compression is obtained which is satisfactory to the engineer. The thickness of the finished binder shall average .... inches and not more than forty (40) per cent. variation from the average thickness specified will be permitted at any one spot. The upper surface of the finished binder shall be parallel to the established grade for the finished pavement. The surface after compression shall show at no place an excess of asphalt cement and any spot showing such excess shall be cut out and replaced with other material. All binder that shows lack of bond or that is in any way defective or which may become broken up before it is covered with wearing surface must be taken up and removed from the street and replaced by good material properly made and laid in accordance with these specifications, at the expense of the contractor. No more binder shall be laid at any one time than can be covered by one day's run of the paving plant on surface mixture. Binder when laid shall be followed and covered with wearing surface as soon as is practicable in order to effect the most thorough bond between the binder and the wearing course. The binder course shall be kept as clean and as free from traffic as is possible under working conditions. If necessary, it must be swept off immediately before laying the wearing surface on it.

No binder shall be laid when in the opinion of the engineer the weather conditions are unsuitable or unless the concrete on which it is to be laid is, even though damp, free from pools of water and has set a sufficient length of time.

13. Requirements.—The finished binder must contain four (4) to seven (7) per cent. of bitumen soluble in cold carbon disulphide, from fifteen (15) to thirty-five (35) per cent. of material passing a 10-mesh screen, and from twenty (20) to fifty (50) per cent. of material passing a one-half (½)-inch screen, the percentage of bitumen to be regulated in accordance with the mesh composition and character of the mineral aggregate of the binder and the percentage of material passing a 10-mesh screen to be regulated in accordance with the traffic conditions upon the street or streets to be paved.

WEARING SURFACE

14. Preparation.—The wearing surface shall be composed of sand, filler and asphalt cement of the character elsewhere herein specified and mixed in proper and definite proportions by weight. The sand and the asphalt cement shall be heated separately to such a temperature as will give, after mixing, a surface mixture of the proper temperature for the materials employed. The sand when used must be at a temperature between 275 and 400°F. The asphalt cement when used must be at a temperature between 250°F. and 350°F. The various ingredients shall be brought together and mixed for at least one minute in a suitable apparatus until a homogeneous mixture is produced in which all the particles are thoroughly coated with asphalt cement. The weights of all materials entering into the composition of the wearing surface shall be verified in the presence of inspectors as often as may be required and the engineer or his representatives shall have access to all parts of the plant at any time.

15. Laying.—The surface mixture prepared in the manner above described shall be brought to the work in wagons covered with canvas or other suitable material and upon reaching the street shall have a temperature between 230°F. and 350°F. The temperature of the surface mixture within these limits shall be regulated according to the temperature of the atmosphere and the working of the mixture and the character of the materials employed. On reaching the street it shall at once be dumped on a spot outside of the space on which it is to be spread. It shall then be deposited roughly in place by means of hot shovels, after which it shall be uniformly spread by means of hot iron rakes in such manner that after having received its final compression by rolling, the finished pavement shall conform to the established grade. The thickness of the finished surface mixture shall average .... inches. Not more than a ten (10) per cent. variation from the average thickness specified will be permitted in any one spot. Before the surface mixture is placed, all contact surfaces of curbs, manholes, etc., must be well painted with hot asphalt cement. After raking, the surface mixture shall at once be compressed by rolling or tamping, after which a small amount of cement shall be swept over it and it shall then be thoroughly compressed by a steam roller weighing not less than two hundred (200) pounds to the inch width of tread, the rolling being carried on continuously at the rate of not more than two hundred (200) square yards per hour per roller, until a compression is obtained which is satisfactory to the engineer. Such portions of the completed pavement as are defective in finish, compression or composition, or that do not comply in all respects with the requirements of these specifications, shall be taken up, removed and replaced with suitable material, properly made and laid in accordance with these specifications at the expense of the contractor. Whenever so ordered by the engineer, a space of twelve (12) inches next to the curb shall be coated with hot asphalt cement, which shall be ironed into the pavement with hot smoothing irons.

No wearing surface shall be laid when in the opinion of the engineer the weather conditions are unsuitable or unless the binder on which it is to be placed is dry. Excessive use of water on the steam roller when compressing the pavement will not be permitted. The finished pavement must be well protected from all traffic by suitable barricades until it is in proper condition for use.

NOTE.—When the pavement is laid alongside of brick or concrete gutters, street car tracks, manhole heads, or liners, it is recommended that the finished surface adjacent to them be left one-quarter (¼) inch high in order to provide for subsequent compression by traffic and to avoid depressions which would otherwise be liable to occur at these points.

16. Requirements.—The finished pavement shall show upon analysis a mesh composition and bitumen contents within the following limits (sieves to be used in the order named):

Bitumen	9.5 to 13.5 per cent.	Total passing 200, 100 and 80-mesh,	Not less than 25 per cent.
Passing 200 mesh	Not less than 10 per cent.		
Passing 80 mesh	10 to 35 per cent.		
Passing 50 mesh	4 to 35 per cent.	Total passing 50 and 40-mesh	15 to 50 per cent.
Passing 40 mesh	4 to 25 per cent.		
Passing 30 mesh	4 to 20 per cent.		
Passing 20 mesh	4 to 12 per cent.	Total passing 30, 20 and 10-mesh	10 to 35 per cent.
Passing 10 mesh	2 to 8 per cent.		
Passing 8 mesh	0 to 5 per cent.		

The minimum amount of bitumen shall be used only in mixtures containing the minimum total passing the 80-mesh. The percentage of bitumen must be increased above the minimum as the total passing the 80-mesh increases. On streets of very light traffic, when the engineer has approved the use of a coarser sand or mixture than that specified for general use, the surface mixture must contain not less than six (6) per cent. of mineral matter passing a 200-mesh sieve and not less than a combined total of eighteen (18) per cent. passing the 200, 100 and 80-mesh sieves. The maximum amount of 200, 100, and 80-mesh material will be regulated according to the kind of sand and asphalt used and the traffic upon the street on which the pavement is to be laid, subject to the maximum requirements elsewhere herein specified under sand and filler.

The above limits as to mesh composition and per cent. of bitumen are intended to provide for such permissible variations as may be rendered necessary by the raw materials used and by the character of the work to be done. The composition of the wearing surface may be varied within the limits above specified at the discretion of the engineer, depending upon the kind of sand, filler and asphalt used and the traffic conditions upon the street or streets to be paved.

CONDITION AT EXPIRATION OF GUARANTEE

17. In addition to the proper maintenance of the pavement during the period of guarantee, the contractor shall, at his own expense, just before the expiration of the guarantee period, make such repairs as may be necessary to produce a pavement which shall:

(*a*) Have a contour substantially conforming to that of the pavement as first laid and free from depressions of any kind exceeding one-half (½) of an inch in depth as measured between any two points three (3) feet apart on a line conforming substantially to the original contour of the street.

(*b*) Be free from cracks or depressions showing disintegration of the surface mixture.

(*c*) Contain no disintegrated surface mixture.

(*d*) Not have been reduced in thickness more than three-eighths (⅜) of an inch in any part.

(*e*) Have a foundation free from such cracks or defects as will cause disintegration or settling of the pavement or impair its usefulness as a roadway.

REPAIRING

18. Repairs, except as provided for below, shall in all cases be made by cutting out the defective binder and wearing surface down to the concrete and replacing them by new and freshly prepared binder and wearing surface made and laid in strict accordance with these specifications.

Whenever any defects are caused by the failure of the foundation, the pavement, including such foundation, shall be taken up and relaid with freshly prepared material made and laid in strict accordance with these specifications.

In all cases the surface of the finished repair shall be at the grade of the adjoining pavement and in accordance with the contour of the street.

The surface-heater method of repairing may be used only in those cases where the repairs are not rendered necessary by:

(*a*) Failure of concrete.

(*b*) Failure of the binder.

(*c*) Failure caused by the disintegration of the lower portion of the wearing surface.

Whenever the surface-heater method is employed, all defective surface shall be removed before replacing it with new material. In all cases the old surface shall be removed to a depth of not less than ¼ in. and the new surface must, when compressed, be not less than

½ in. in thickness. The heat shall be applied in such a manner as not to injure the remaining pavement. All burnt and loose material shall at once be completely removed and, while the remaining portion of the old pavement is still warm, shall be replaced by new and freshly prepared wearing surface made and laid in strict accordance with these specifications.

19. NOTE TO ENGINEERS. Filler.—As portland cement is more expensive than lime dust, the specification should distinctly state which kind of filler is desired.

Binder.—The following clause has been suggested as being descriptive of the practice in some cities. The committee, however, does not feel like recommending it in a general specification. If this clause is incorporated in the specifications it should be clearly stated whether or not the practice described therein will be permitted by the city engineer.

With the permission of the city engineer, not to exceed twenty (20) per cent. of crushed old asphalt surface mixture of suitable character may be used in combination with the binder stone, provided such mixture produces a binder complying in all respects with the requirements of these specifications.

METHODS FOR TESTING AND SAMPLING

The following methods are recommended as being sufficiently accurate for general use. In cases of dispute the standard methods adopted by the American Society for Testing Materials must be employed.

PENETRATION TEST

20. Penetrations shall be taken by means of a penetrometer, which shall be so constructed as to correctly register in one-hundredths of a centimeter the depth to which a Robert's Sharps No. 2 needle will penetrate the sample under examination under a given load without appreciable retarding friction for a given time period.

For penetrations at 77°F. the time period shall be five (5) seconds and the total weight operating on the needle shall be one hundred (100) grams except in the case of flux, where the time period is one (1) second and the total weight fifty (50) grams.

The samples to be tested should preferably be in circular tin boxes about two and one-quarter (2¼) inches in diameter and about three-quarters (¾) of an inch deep (2-ounce Gill style can, obtainable from the American Can Company). Where very soft materials are to be tested or penetrations are to be taken at 100°F. or 115°F., a tin not less than two (2) inches deep and having the same diameter specified above should be used to prevent the needle from striking the bottom of the tin before it has penetrated the sample to the full depth.

All samples shall be melted at a temperature just high enough to render them liquid (250 to 300°F.) and should then be thoroughly stirred until homogeneous and free from air bubbles. After cooling sufficiently in the air at laboratory temperature they must be immersed for at least thirty (30) minutes in water maintained at the temperature at which the test is to be made (77°F.). During testing the sample shall be accurately maintained at the temperature specified.

The average of from three (3) to five (5) tests, which must not differ more than five (5) points (five hundredths [0.05] of a centimeter) between maximum and minimum, shall be taken as the penetration of the sample, the needle being wiped off with a dry cloth after every determination.

Remarks.—This test measures the consistency of the material under examination. The limits of accuracy of this test may be considered as being within five (5) per cent. of the reading obtained (above or below).

DUCTILITY TEST

21. This test is usually first made on the asphalt cement itself. If this fails to show the required ductility, the pure bitumen must be extracted and tested. The proper methods for obtaining the pure bitumen vary with the asphalt being examined and are too lengthy for description here. (See *Proc. Am. Soc. Test. Mat.*, **9**, 594.)

Preparation of Briquette.—The molding of the briquette may be done as follows:

The mold should be placed upon a brass plate. To prevent the asphalt from adhering to the plate and the inner side of the two removable pieces of the mold, they should be well amalgamated. The different pieces of the mold should be held together in a clamp or by

means of an India rubber band. The material to be tested is poured into the mold while in a molten state, a slight excess being added to allow for shrinkage on cooling. After the briquette is nearly cool, it is smoothed off level by means of a heated palette knife. When cooled, the clamp is taken off and the two side pieces removed, leaving the briquette of asphalt firmly attached to the two ends of the mold, which thus serve as clips. The briquette should be immersed in water maintained at the required temperature for at least thirty (30) minutes or until the whole mass of bitumen is at 77°F. It is then pulled apart at the required rate of speed in a suitable machine, the briquette being entirely immersed in water maintained at 77°F. during the entire operation of pulling. Any pieces of dirt, wood, or extraneous matter in the briquette may cause the fracture of the fine thread before the true maximum ductility of the material under examination has been reached. Great care should be observed, therefore, to avoid the presence of such foreign matter in the bitumen when it is poured into the mold. The average of at least two tests shall be recorded as the ductility of the sample under examination. These tests must not differ more than twenty (20) per cent. from their average.

Remarks.—This test measures approximately the cementing value of a bitumen, but it is not necessarily a measure of the relative cementing value of different bituminous materials or the same bituminous material at different penetrations. The limits of accuracy of this test may be considered as being within twenty (20) per cent. of the reading obtained (above or below).

DETERMINATION OF TOTAL BITUMEN IN REFINED ASPHALTS AND ASPHALT CEMENTS

22. One to two grams of the sample shall be weighed into a tared 200-c.c. wide-mouth Erlenmeyer flask and covered with 100 c.c. of chemically pure carbon disulphide. Agitate until all lumps appear and nothing adheres to the bottom of the flask. Cork and allow to stand fifteen (15) minutes. Filter off on a Gooch crucible with asbestos felt or a weighed filter paper and wash until the washings come through practically colorless. Dry the flask and filter at 250°F. Evaporate the filtrate containing the bitumen, burn to an ash and add to the residue on the filter.

Remarks.—The limits of accuracy of this test as applied to bitumens containing considerable proportions of non-bituminous matter may be considered as being within one-half (½) per cent. above or below the result obtained. In practically pure bitumens one-quarter (¼) per cent. above or below is the ordinary limit of accuracy.

DETERMINATION OF BITUMEN SOLUBLE IN CARBON TETRACHLORIDE

23. One gram of the sample shall be weighed into a tared 200-c.c. wide-mouth Erlenmeyer flask and covered with 100 c.c. of chemically pure carbon tetrachloride. Agitate until all lumps disappear and nothing adheres to the bottom of the flask. Cork and allow to stand eight (8) hours in the dark. Filter off on a Gooch crucible with asbestos felt or a weighed filter paper and wash until the washings come through practically colorless, using not less than 100 c.c. of fresh solvent. Dry the filter at 250°F.

Remarks.—The amount of bitumen insoluble in carbon tetrachloride is indicative of whether or not decomposition has been produced by improper heat treatment. The limits of accuracy of this test may be considered as being within one-half (½) per cent. above or below the result obtained.

VOLATILIZATION TEST

24. Twenty (20) grams of the sample shall be placed in a weighed tin box, two and one-quarter (2¼) inches in diameter and three-quarters (¾) of an inch high (two-ounce Gill style can, obtainable from the American Can Company), and heated five (5) hours at 325°F. The heating shall be done in a ventilated oven which shall have reached the temperature specified before the introduction of the samples and which is maintained within two (2) degrees of that temperature throughout the test. The tin can should be insulated by a sheet of asbestos or other material from direct metallic contact with the sides or walls of the oven. The bulb of the thermometer should be immersed in a control bath immediately alongside of the sample being tested, the container and the method of insulation being the same in both cases.

Remarks.—This test indicates the extent to which bitumens in the course of time lose their more volatile hydrocarbon constituents and the hardening resulting from volatiliza-

tion and chemical change. It may be considered as an accelerated exposure test. The limits of accuracy of this test cannot be definitely stated, owing to the widely varying results obtained by the use of different types of ovens and failure to carefully observe all the conditions prescribed. When carefully conducted according to the above directions, a test showing six (6) per cent. loss should be considered as passing a specification calling for not over five (5) per cent. loss.

FLASH TEST

25. Flash test shall be made in a circular tin can about two and one-quarter (2¼) inches in diameter and about one and three-eighths (1⅜) inches deep (3-ounce Gill style, American Can Company), provided with a suitable transparent cover of mica, or glass, etc. This cover shall be provided with two apertures for the insertion of the thermometer and test flame. The aperture for the thermometer shall be three-eighths (⅜) of an inch in diameter and shall be centrally located. The aperture for the test flame shall be triangular in shape measuring one-half (½) inch on the base and three-quarters (¾) of an inch in height. The base shall coincide with the rim of the can. A thermometer approximately fifteen (15) inches long, graduated in single degrees, shall have its bulb completely immersed in the material being tested. It shall not touch the bottom of the can and shall be suspended in the proper position. The can shall be filled with the material to be tested so as to leave a one-half (½) inch vapor space when melted. The material shall be heated at the rate of 10°F. a minute and the test flame applied every 5°F. after a temperature 300°F. has been reached. No correction for emergent stem shall be made. The test flame shall be one-eighth (⅛) of an inch long and shall be dipped in just below the surface of the cover and then immediately withdrawn.

Remarks.—This test indicates the temperature at which inflammable vapors are given off in an enclosed space. It supplements the volatilization test and guards against the use of a material containing too large an amount of volatile hydrocarbons. The limit of accuracy of this test may be considered as being five (5) degrees above or below the reading obtained.

SPECIFIC GRAVITY TEST

26. (*a*) Fluid Materials:

The specific gravity of fluid materials shall be taken in the usual way in a pycnometer at 77°F.

(*b*) Viscous Fluid and Semi-Solid Materials:

The specific gravity of these materials shall be taken in a cylindrical weighing bottle pycnometer as given on page 10, Bulletin No. 38, of the Office of Public Roads. (See page 127.)

(*c*) Hard Solid Materials:

The specific gravity of hard, solid materials shall be taken by the displacement method

DETERMINATION OF BITUMEN CONTENTS AND MESH COMPOSITION OF BINDER

27. Weigh out from 350 to 500 grams of the binder and extract the bitumen from it in a centrifugal extractor or suitable continuous hot extractor, using chemically pure carbon disulphide as a solvent for the bitumen. Follow the same general method for the drying and sifting of the mineral aggregate as described in the method for analyzing surface mixtures. The sieves to be used are as follows:

1½-in., 1-in., ¾-in. and ½-in. circular opening, and 10-mesh.

Remarks.—The limits of accuracy of this test are as follows:

For bitumen contents, three-tenths (0.3) per cent. above or below the result obtained. For mesh composition, ten (10) per cent. of the result obtained (above or below).

DETERMINATION OF BITUMEN CONTENTS AND MESH COMPOSITION OF SURFACE MIXTURES

28. The sample of surface mixture should be heated to about 300°F. until soft and ten to twenty grams of it weighed on to a tared S. & S. filter paper No. 595, 11 cm. in diameter. The filter paper and contents should be placed in a funnel and washed with chemically pure carbon disulphide until the washings run through practically colorless. Dry the filter paper and residue at 250°F. for one-half (½) hour. Open the filter paper carefully and remove the mineral aggregate. Scrape off the dust adhering to the paper as thoroughly as possible

with a blunt palette knife and add it to the mineral aggregate. Evaporate the filtrate containing the bitumen, burn the bitumen, add the filter paper to it and burn to an ash. Add the ash to the mineral aggregate previously removed from the filter paper and weigh. The difference between the weight of surface mixture originally taken and the combined weight of the ash and residue is considered as the weight of bitumen in the sample. This combined ash and residue is then sifted through the following sieves (in the order named) and the percentages of the various sized particles calculated:

200, 100, 80, 50, 40, 30, 20, 10 and 8.

Sifting shall be continued on each sieve until less than one (1) per cent. passes through the sieve during the last minute of sifting.

If desired, the surface mixture may be extracted in a centrifuge or in any suitable form of extractor with hot chemically pure carbon disulphide and the combined ash from the extracted bitumen and the mineral aggregate sifted as above.

Remarks.—The limits of accuracy of this test are as follows:

For bitumen contents, three-tenths (0.3) per cent. above or below the result obtained. For mesh composition, ten (10) per cent. of the result obtained (above or below).

SAMPLES

29. Samples should be put in clean, dry containers, preferably tin boxes or cans. The following amounts of the different materials are required for test:

Binder stone	5 lb.
Filler	½ lb.
Sand	1 lb.
Refined asphalt	1 lb.
Asphalt cement	1 lb.
Flux	1 lb.

Method of Sampling.—Extreme care should be taken in every case to obtain a sample which is truly representative of the material to be examined. These samples are for the use of the testing laboratory only and should not be used for testing at the plant before submitting them to the laboratory. The particular precautions to be observed in each case are given below:

Binder Stone.—A sufficient number of 5-lb. samples to be taken from different parts of the pile. These should be thoroughly mixed together and reduced by quartering to the desired size.

Filler.—A sample should be taken from several bags and mixed.

Sand.—Samples should be taken from the interior of the pile where the sand is damp. A sufficient number of 1-lb. samples to be taken from different parts of the pile. These should be thoroughly mixed together and reduced by quartering to the desired size.

REFINED ASPHALT AND ASPHALT CEMENT

30. In Barrels.—At least one sample should be taken from each batch. It should be taken at sufficient depth below the surface to insure obtaining representative material free from all dirt or other extraneous matter, and at a point not less than four (4) inches distant from the top and sides of the barrel.

In Tank Cars.—The contents of the tank should be heated until completely liquid throughout. It should then be agitated and thoroughly mixed by means of air or steam, after which the sample shall be taken from the dome in such a manner as to obtain the asphalt from a point at least three (3) feet below the surface.

In Kettles.—The contents of the kettles must be completely liquid and thoroughly agitated previous to and during sampling. The sample may be taken from the pipe through which the material is delivered to the mixer or by means of a clean dipper.

31. Flux.—The directions given for sampling refined asphalt and asphalt cement apply to this material except that under ordinary conditions it is not necessary to agitate the contents of the tank car.

32. Surface and Binder Mixtures.—Samples should preferably be taken on the street after the mixture has been shoveled and raked. Samples taken from the plant shall be obtained from the wagons, special care being observed to avoid material from the top of the

load or which appears to vary from the average. Samples should be pressed between a sheet of paper and trimmed while hot to a convenient size.

REVISIONS OF SPECIFICATIONS FOR SHEET ASPHALT PAVING

Edition of 1916, as Adopted in 1918

Page 397.—

(1) Section 4, first paragraph, first line:
Insert the words "*and asphalt cements*" after the words "The refined asphalts."

(2) Section 4, first paragraph, third line:
Insert the words "*and mixing*" after the words "methods of refining."

(3) Section 4, second paragraph, first line:
Insert the words "*and asphalt cements*" after the word "asphalts."

(4) Section 4, second paragraph, fourth line:
Insert the words "*and asphalt cement*" after the word "asphalt."

Page 397.—

(5) Section 4, second paragraph, sixth line:
Delete the word "foregoing."

Page 398.—

(6) Section 8, first paragraph:
Rewrite as follows: "*Samples.* If the contractor desires to use an asphalt cement prepared at the refinery he must submit with his bid a one (1) pound sample of such asphalt cement, together with a statement as to the source and character of the material used in its manufacture. If the contractor intends to prepare the asphalt cement at his plant by mixing together refined asphalt and flux, he must submit with his bid one (1) pound samples of the refined asphalt, flux and asphalt cement that he proposes to use, together with a statement as to the source, character and proportions of the materials composing them. No contract shall be awarded to any bidder whose samples do not comply in every respect with these specifications. No asphalt other than that specified in his bid shall be used by any contractor except with the written consent of the engineer and provided that it complies in all respects with the requirements of these specifications."
(Follow with the second paragraph as it now stands.)

Page 398.—

(7) Section 9, first paragraph:
Rewrite as follows: "The asphalt cement shall be prepared—
(*a*) By the careful refining of natural liquid mineral bitumens to the proper penetration.
(*b*) By fluxing hard asphalt or combinations of asphalt with flux."

(8) Section 9, second paragraph, first line:
Commence the paragraph with the words "*In preparing fluxed asphalt cements.*"
(Follow with the paragraph as it now stands.)
NOTE.—The above changes are to more clearly provide for the use of asphalt cements prepared at the refinery and not at the paving plant.

Page 398.—

(9) Section 7:
Change the sand specifications with respect to 8 and 10 mesh materials so that they will read as follows:
Passing 10 mesh and retained on 20 mesh, 4 to 15 per cent.
Passing 8 mesh and retained on 10 mesh, 0 to 8 per cent.

Page 401.—

(10) Section 16:
Change the requirements for the mesh composition of the finished pavement to accord with the above change in sand, as follows:
Passing 10 mesh, 2 to 12 per cent.
NOTE.—The above changes permit the use of a slightly coarser sand than that allowed under the present specifications and tend to increase the stability of the pavement.

(11) Page 397, Section 5, Clause "c;"

(12) Page 399, Section 10, Clause "d;"

(13) Page 403, Section 24:
Change the amount of material used in the volatilization test from 20 to fifty (50)

grams and the loss permitted from 5 per cent. to three (3) per cent., and the depth of the box to 1½ in.

NOTE.—This will bring the test more nearly in accord with A. S. T. M. Standard method, and will leave the standard of quality substantially as at present.

(14) Page 399, Section 10, Paragraph "a:"
Add the words "*and free from water.*"

(15) Page 403, Section 22, third line:
Change the word "appear" to "disappear."

(16) Page 403, Section 23, fourth line:
Change the word "eighteen" to "*eight.*"

Specifications for Surface Treatments with Cold Bituminous Materials, Adopted by the American Society of Municipal Improvements

SPECIFICATIONS FOR SURFACE TREATMENTS WITH COLD BITUMINOUS MATERIALS

1. Description of Bituminous Surface Treatment.—The bituminous surface treatment shall consist of one application of refined tar or cut-back asphalt cement covered with a thin layer of top dressing as hereinafter specified.

NOTE.—This specification provides for a surface treatment which may be used on thoroughly cleaned broken stone or gravel roads in good condition; for the re-treatment of tar surfaces or the maintenance of seal coats on tar-macadam or tar-concrete pavements if refined tar is used for the surface treatment; or for the re-treatment of asphaltic surfaces or the maintenance of seal coats on asphaltic macadam or asphaltic concrete pavements if cut-back asphalt cement is used for the surface treatment.

2. Refined Tar and Cut-Back Asphalt Cement.—The refined tar or cut-back asphalt cement, hereinafter referred to as bituminous material, used in the construction of the surface treatments shall conform with its respective specification covering the chemical and physical properties of bituminous materials included under the item entitled "Refined Tar and Cut-Back Asphalt Cement for Cold Surface Treatment."

3. Preparation of Surface of Roadway.—After the surface of the roadway shall have been repaired, is well consolidated, and free from holes or depressions, and when thoroughly dry, it shall be swept clean of all dust, dirt or other loose material with horse- or power-drawn brooms and bass or other fine-fiber brooms, or with stiff-fiber hand brooms and bass or other fine-fiber brooms. Newly constructed broken stone or gravel roads shall be subjected to traffic for at least one month before the construction of the bituminous surface thereon.

4. Application of Bituminous Material.—After the surface shall have been cleaned, and when thoroughly dry, the unheated bituminous material shall be uniformly applied to the prepared surface of the roadway by means of a pressure distributer as hereinafter specified, and in accordance with the directions of the engineer. The total amount of bituminous material to be used in the surface treatment shall be applied in one application and shall not be less than one-eighth (⅛) nor more than one-half (½)

gallon per square yard, the precise quantity being determined by the engineer.

NOTES.—The first treatment of broken stone or gravel roads should consist of an application of between one-third (⅓) and one-half (½) gallon per square yard. Generally, subsequent treatments on the above types of roads, and bituminous pavements, should be less than one-third (⅓) gallon per square yard.

5. Pressure Distributer.—The pressure distributer employed shall be so designed and operated as to distribute the bituminous material specified uniformly under a pressure of not less than twenty (20) pounds nor more than seventy-five (75) pounds per square inch in the amount and between the limits of temperature specified. It shall be supplied with an accurate pressure gage so located as to be easily observed by the engineer while walking beside the distributer. It shall be so operated that, at the termination of each run, the bituminous material will be at once shut off. It shall be so designed that the normal width of application shall be not less than six (6) feet and so that it will be possible on either side of the machine to apply widths of not more than two (2) feet. The distributer shall be provided with tires of widths dependent upon the following relationship between the pressure per square inch of tire and the diameter of the wheel; for a two- (2) foot diameter wheel, five hundred (500) pounds shall be the maximum pressure per linear inch of width per wheel, an additional pressure of thirty (30) pounds per inch being allowed for each additional three (3) inches in diameter.

6. Application of Top Dressing.—Immediately after the completion of the application of the bituminous material, from fifteen (15) to twenty-two (22) pounds of broken stone chips, pea gravel, grit or other mineral matter which will meet the following requirements, shall be uniformly spread, as directed by the engineer, over the surface of the bituminous material.

7. The broken stone, or pea gravel, grit or other mineral matter shall consist of clean, hard, tough, durable material, free from soft or disintegrated particles, dirt, or other objectionable matter.

8. When tested by means of laboratory screens it shall meet the following requirements:

Passing ½-in. screen, not less than..... 85 per cent.
Retained on ¼-in. screen, not less than..... 85 per cent.

9. Seasonal and Weather Limitations.—No bituminous material shall be applied when the air temperature in the shade is below 10°C. (50°F.), except by the written permission of the engineer.

10. Measurement and Payment.—The quantity of surface treatment to be paid for under this item shall be the number of square yards, measured horizontally, satisfactorily completed in accordance with the specifications. The price stipulated in this item shall include the furnishing and screening of the top dressing, the preparation of the roadway surface, the application of the bituminous material, and the spreading of the top dressing, and all materials, work and expenses incidental to the completion of the surface treatments, except the furnishing of the bituminous material, which will

be included for payment under the item entitled "Refined Tar and Cut-Back Asphalt Cement for Cold Surface Treatment."

SPECIFICATIONS FOR REFINED TARS AND CUT-BACK ASPHALT CEMENTS FOR COLD SURFACE TREATMENTS

11. Previous Service.—The contractor will be required to show to the satisfaction of the engineer, that the company manufacturing the bituminous material he proposes to use under a given specification has, for a period of at least two years, manufactured bituminous material in a thoroughly equipped plant, and that the bituminous material which he proposes to use shall have been in continuous and successful use in the surface treatment of roadways for a period of at least two years previous to the date of the letting on which his proposal was submitted.

12. Refined Tar.—Its specific gravity at 25°/25°C. (77°/77°F.) shall not be less than 1.100 nor more than 1.180.

13. When tested by means of the Engler viscosimeter at 40°C. (104°F.), the specific viscosity of the first 50 c.c. passing the orifice of the viscosimeter shall be not less than 10 nor more than 35, with a maximum range not exceeding 5.

NOTE.—Low specific viscosities should be used for work in the northern States and high specific viscosities for work in the southern States.

14. Its bitumen, as determined by its solubility in chemically pure carbon disulphide at room temperature shall not be less than 90 per cent.

15. When distilled according to the Am. Soc. Test. Mat. Standard Method, it shall yield not more than 5 per cent. of distillate at a temperature lower than 170°C. (338°F.); not more than 30 per cent. shall distil below 270°C. (518°F.); and not more than 40 per cent. shall distill below 300°C. (572°F.).

16. Cut-Back Asphalt Cement.—The cut-back asphalt cement shall consist of a mixture of asphalt cement and naphtha, which will comply with the following specifications:

17. Its specific gravity at 25°/25°C. (77°/77°F.) shall be not less than 0.890.

18. When 20 grams of the material are maintained at a uniform temperature of 163°C. (325°F.) for 5 hours, in an open cylindrical tin dish 5½ cm. (about 2¼ in.) in diameter, with vertical sides measuring approximately 3½ cm. (about 1½ in.) in depth, the loss in weight shall be not less than 30 per cent. nor more than 40 per cent.

19. When tested with a standard No. 2 needle under a load of 100 grams for 5 seconds at 25°C. (77°F.), the penetration of the residue, expressed in hundredths of a centimeter, shall be between 50 and 85.

20. When tested by means of the Engler viscosimeter at 25°C. (77°F.), the specific viscosity of the first 50 c.c. passing the orifice of the viscosimeter shall be not less than 25 nor more than 35.

21. When distilled according to the Standard Method of the Am. Soc. Test. Mat., the specific gravity of the distillate below 300°C. (572°F.) shall be not less than 0.730 nor more than 0.780.

22. Its bitumen, as determined by its solubility in chemically pure carbon disulphide at room temperature, shall be not less than 99.5 per cent.

23. Delivery.—The bituminous material shall be delivered in suitable containers, far enough in advance of its use in the work to permit the necessary tests to be made. Each container shall be plainly labeled with the trade name of the bituminous material, name of manufacturer, gross weight and net weight. Each shipment and each carload shall be kept separate.

24. Bills of Lading.—The contractor shall furnish the engineer bills of lading, or correct copies thereof, which shall state the trade name of the bituminous material and the name and address of the company manufacturing and supplying it.

25. Samples.—Samples will be taken by the engineer from each carload of bituminous material when delivered at the work, unless satisfactory arrangements can be made for sampling before shipment. Such samples shall be analyzed by the engineer to assure the delivery of a bituminous material of the specified quality.

26. Work Included.—Under this item the contractor shall furnish and deliver on the work, at such points as directed, bituminous material which conforms with the specifications of either refined tar or cut-back asphalt cement, as specified.

27. Measurement and Payment.—The quantity of bituminous material to be paid for under this item shall be the number of gallons placed on the highway in accordance with the specifications and requirements, or used as directed for other purposes. Bituminous material that is wasted by the contractor shall not be included in the measurement under this item. The price stipulated in this item shall include the cost of furnishing, hauling and delivering the bituminous material on the work, and all expenses incidental thereto.

Specifications for Asphaltic Concrete (Including "Bitulithic" and Specifications for Coal Tar and Water-Gas Tar), Adopted October 12, 1916, by the American Society of Municipal Improvements

SPECIFICATIONS FOR BITUMINOUS CONCRETE PAVEMENT

Sub-grade.—The contractor will be required to do all of the grading necessary to bring the surface to the proper sub-grade as determined by the lines and grades given by the engineer. If the material at sub-grade is of an unstable character and unfit for foundation, the contractor shall make such additional excavation as may be determined by the engineer and refilled with approved material. After all necessary grading has been done to bring this surface to sub-grade, the street shall be thoroughly rolled with an approved road roller weighing not less than ten tons. If settlement occurs the depressions shall be filled and then re-rolled until the surface is solid, uniform and parallel with the grade and cross-section of the finished pavement. All filling shall be free from animal or vegetable matter and of a character approved by the engineer. In case of spongy or yielding sub-grade some other means besides ordinary rolling and sprinkling must be employed to obtain satisfactory compaction of the sub-grade. In case of loose, sandy soils, a small amount of cinders, gravel or fine crushed stone spread over the surface will often put it in a condition to be compacted under the roller. In the case of clay soils that puddle up and wave or creep under continued rolling, it is best to roll as dry as possible and to be sparing in the use of water when rolling the first layer of macadam. Cinders, gravel or stone screenings will often help in rolling such sub-grades.

Sub-drainage.—When the soil is of such a character that it retains an excessive amount of moisture, such as clay, subject to swelling or heaving under the action of frost, or sands similar to quicksand that do not afford a ready natural drainage, sub-drains should be provided.

These may be of two general kinds: first, tile drains of porous material or of vitrified tile laid with open joints; second, trenches filled with broken stone, gravel, cinders or other similar material.

In some cases it may be sufficient to construct a sub-drain on each side of the roadway at or near the lines of the gutters, but when the soil is of a very wet nature it may be advisable to lay additional lines of drains which may be in or near the middle of the roadway. This system of drains may be varied by diagonal lines of drains running from near the crown of the roadway to the gutters.

In all cases the drains should have connections with the existing sewers, catch basins or inlets.

New Macadam Foundation.—If the pavement is to be laid on a new macadam foundation or base, the latter shall be built as follows:

The total thickness of the macadam base will vary according to character of soil, drainage, kind of stone available, etc. In general, the macadam base should be constructed of broken stone which is sound, hard and durable under traffic. The broken stone should be separated into different sizes by screening, the smaller sizes with the dust being used to fill and bond together the larger sizes. The thickness of the base should be regulated by experience in constructing ordinary water-bound macadam roads in similar situations, the total thickness of pavement, including wearing surface, being made the same or a little less than well constructed macadam.

After the sub-grade has been carefully prepared, spread a layer of clean stone passing a three (3) to three and one-half (3½)-inch revolving screen and held on a two (2)-inch screen to a depth sufficient when thoroughly rolled to form about two-thirds (⅔) of the total thickness of the base. The thickness of this layer should be regulated by laying on the sub-grade at proper intervals, cubical blocks of wood of the proper dimensions to give the desired thickness. Over this layer of stone, spread with shovels stone screenings in sufficient quantity to fill the voids between the larger stone. The screenings should be spread gradually and thoroughly rolled with a road roller weighing at least 10 tons during the process of spreading the screenings. As the screenings are worked into the coarse stone under the roller, more should be added here and there where voids appear. At first the rolling should be done dry until the stone appears to be well filled, then the surface should be well sprinkled and again rolled, the rolling and sprinkling continued until the layer of stone is thoroughly compacted and no more screenings can be forced in. Just enough screenings shall be used to fill and bond the stone, leaving no surplus screenings on the top.

The above method may be varied by using the crusher run of stone without the addition of any other filler where the small sizes are not in excess. Also a filler other than stone screenings, such as bank gravel or sand, may be used in some cases where experience with materials available shows better results can be obtained. Under some conditions the character of soil and stone available may be such as not to require the use of any filler with the stone of the first course. The specifications given, however, represent the best average practice where stone with bonding value, such as limestone or trap rock, can be obtained.

When the first layer of macadam is completed as specified, spread a second layer of clean stone passing a two (2) or two and one-half (2½)-inch screen and held on a one (1)-inch screen to a depth sufficient when thoroughly rolled to form the remaining one-third of the total thickness of the base. Over this layer of stone spread evenly with shovels stone screenings and roll with the application of water by sprinkling. The sprinkling and rolling shall be continued until the stone is well bonded and until no more compression can be observed under the roller. Just enough filler should be used to accomplish this purpose and not enough to form a layer or film over the surface of the stone. It is better not to fill the stone quite flush, leaving the coarse particles of stone slightly projecting, so as to have a coarse, grainy base upon which to put the wearing surface.

As an alternate method of construction the macadam may be well filled with screening, watered and rolled until flushed up smooth. Over the surface of the macadam base thus constructed shall be spread a third layer of clean stone of a size to pass a two (2)-inch ring and be retained on a one (1)-inch ring. This layer of stone should average one (1) inch after rolling or practically only one stone deep and is for the purpose of forming a binder or key between the base and wearing surface and thus preventing lateral displacement of the surface. After being spread evenly it shall be lightly rolled only enough to partially imbed the stone and set them firmly in place without crushing or forcing sufficient fine material up from below to fill the surface voids entirely. The final rolling should be done while the macadam base is still moist and comparatively soft.

The thickness of the pavement, including base and wearing surface, should vary according to local conditions and should be fixed by the engineer in charge when all the varying conditions of soil, drainage, traffic and materials of construction are understood. In general, a thickness of macadam base of eight (8) inches with a wearing surface of two (2) inches will be enough for any except the most adverse conditions, and a base of four (4) to five (5) inches with a wearing surface of from one and one-half (1½) to two (2) inches will meet the most favorable conditions of firm, unyielding soils and light traffic.

Old Macadam Foundation.—If the pavement is to be laid on an old macadam foundation, the surface shall be thoroughly swept and cleaned of all fine material that may be caked upon the surface of the stone or lying loose as dust, thereby exposing the clean, coarse stone for the reception of the bituminous concrete.

If the old macadam does not present the desired coarse, grainy surface, or is not at proper and satisfactory grade after cleaning, it shall be spiked up and redressed to the desired crown and grade, the coarse stone being brought to the top by harrowing or otherwise, or new stone added where needed. It shall then be watered and rolled until thoroughly compacted. If the result is not the required coarse, grainy surface, a layer of clean stone shall be spread and lightly rolled as described above in the paragraph relating to new macadam foundation.

Concrete Foundation.—When a portland cement concrete foundation is used, it should be laid according to the standard specifications adopted for concrete foundation. The surface, however, should be roughened to form a key for the wearing surface. This may be done by using coarse stone of fairly uniform size and laying the concrete fairly wet, or by brooming, washing with a hose before hard set, tamping with grooved rammers, or by spreading a light layer of coarse, clean stone over the fresh concrete and lightly tamping.

Curb.—If a curb or curb and gutter is used, the face against which the paving material will be laid shall be painted with a coat of hot bituminous cement in advance of the pavement.

A curb or curb and gutter should be used in all cases of a street devoted to miscellaneous uses and where there is occasion for many vehicles to stop at the curb line; but in the case of a park driveway or a suburban highway, a bituminous concrete pavement may be successfully used without a curb. No other protection for the edge of the pavement is required except to provide a coarse, grainy base into which the paving material is rolled.

Wearing Surface.—On the foundation, as heretofore specified, shall be laid the bituminous concrete wearing surface, which shall consist of a mineral aggregate mixed with bituminous cement and laid as hereinafter specified.

This wearing surface shall have a thickness of .... inches after thorough compression with a roller.

For heavy traffic a thickness of two (2) inches is sufficient for all practical purposes and in some cases will afford more stability than a greater thickness.

For moderate and light traffic one and one-half (1½) inches will be sufficient thickness for the wearing surface when laid on a well constructed base as specified above.

Mineral Aggregate.—The mineral aggregate shall consist of a mixture of broken stone and sand, to which shall be added, as required, stone, dust or portland cement

Any sound, durable stone, either trap rock, limestone or granite, usually considered suitable for macadam, may be used. It should be broken as nearly cubical as practicable. It should not show distinct planes of cleavage or crystalline faces and should not readily crush or split under the roller when being rolled in the pavement. Between two kinds of stone, choice should generally be made of the one showing the greater toughness rather than hardness. A certain percentage of absorption, such as is shown by the better grades of limestone, is a desirable quality, as the bonding strength of the cement is somewhat improved thereby.

The sand shall be hard-grained, moderately sharp, free from loam or other foreign material and varying in size from that passing a one-quarter (¼)-inch screen to dust passing a 200-mesh screen, and there should not be over ten (10) per cent. held on the 10-mesh screen, nor over five (5) per cent. passing the 200-mesh screen.

Dust in the form of finely ground stone or portland cement may be added to the mixture, but in such quantities that the screenings of the total ingredients entering into the mix shall in no case show over ten (10) per cent. by weight passing a 200-mesh screen.

The proportions of the various ingredients composing the Bituminous Concrete shall be as follows:

Bitumen	7– 9 per cent.
Passing 200 mesh screen	7–10 per cent.
Passing 80 mesh screen, but retained on a 200	10–20 per cent.
Passing 40 mesh screen, but retained on an 80	10–25 per cent.
Passing 20 mesh screen, but retained on a 40	10–25 per cent.
Passing 8 mesh screen, but retained on a 20	10–20 per cent.
Passing 4 mesh screen, but retained on an 8	15–20 per cent.
Passing 2 mesh screen, but retained on a 4	5–10 per cent.

The minimum amount of bitumen allowed shall be used only in mixtures containing the minimum total passing the 80-mesh. The percentage of bitumen must be increased above the minimum as the total passing the 80-mesh increases.

The item designated as dust includes, in addition to the stone dust or portland cement that may be added, such fine sand passing a 200-mesh screen as may be found self-contained in the sand to be used, and such 200-mesh mineral as may be self-contained in the refined asphalt.

Method of Mixing.—The aggregate shall be dried and heated in properly designated driers before mixing with the bituminous cement. The driers shall be of the revolving type, thoroughly agitating and turning the materials during the process of drying. When the aggregate is thoroughly dried and heated to a temperature of from 200 to 350°F., depending upon the bituminous cement used, it shall be immediately before cooling or exposure to moisture, mixed with the hot bituminous cement as hereinafter specified. If stone dust is used, it shall be introduced directly into the mixer without passing through the drier.

The bituminous cement shall be melted in a tank arranged so the heat can be properly and easily controlled and regulated. When melted and raised to a temperature of from 200 to 350°F., depending on the bituminous cement used, it shall be combined in the proper proportions with the hot aggregate and immediately mixed in a properly designed revolving mixer until a thorough and intimate mixture of the ingredients has been accomplished and the particles composing the aggregate are evenly and thoroughly coated with the bituminous cement. The mixer shall not be exposed directly to the action of fire.

Method of Laying.—While still hot from the mixer, the paving mixture shall be spread evenly on the foundation with hot iron rakes and shovels, so that when compressed with the roller it shall have the thickness specified, with the surface even and true to grade. Along the curb and around manholes, catch basins and other obstructions in the street, where the roller cannot reach, the compression shall be secured by the use of hot iron tampers. The rolling and tamping shall be done as quickly as possible after the material is spread, while still hot and pliable. When the paving mixture is hauled on the street in dump wagons it shall be, when ordered by the engineer, kept covered with canvas to retain heat, dumped on platforms and shoveled into place and raked to the proper grade. As soon as spread the paving mixture shall be rolled with a tandem roller weighing at least six (6) tons and the rolling continued, working lengthwise and diagonally of the street. When practicable, additional compression in the wearing surface should be secured by the use of a ten (10)-ton roller. Rolling must be steadily kept up and continued until all roller marks shall disappear and the surface gives indications of no further compressibility.

The paving shall be done continuously, so the number of joints between the hot and cold material may be reduced to the minimum. When it is not practicable to lay it continuously and a joint is unavoidable, the edge of the cold material shall be trimmed down to a rough feather edge, and the surface, where the joint is to be made, painted over with bituminous cement, the hot material raked over the feathered edge and thoroughly rolled. Instead of trimming the cold material, joint strips may be used consisting of strips of canvas about eighteen (18) inches wide with three parallel lines of three-quarter (¾)-inch ropes sewed on the under side about three (3) inches apart. The joint strips shall be laid on the feather edge of the freshly raked material with the upper rope at the line where the thickness begins to decrease and the rolling completed on top of the canvas as for finished pavement. The faces of the curb and gutter, iron castings, etc., shall be painted with bituminous cement before the paving mixture is laid.

Surface Finish.—As soon as possible after the rolling of the mixture is finished, and while the surface is still fresh and clean, and, if possible, while warm, a seal coat of bituminous cement of proper consistency to be flexible when cold shall be spread over the surface. It shall be applied while at a temperature of from 200° to 350°F., depending upon the bituminous cement used, and evenly spread with rubber squeegees or mops. Only a sufficient coat

shall be spread to flush the surface voids without leaving an excess. Immediately over this, a top dressing of torpedo sand, fine gravel or stone chips free from dust, which must be thoroughly dry and heated in cold weather, shall be spread and thoroughly rolled into the surface. A small surplus shall be left to be worn in or worn away by the traffic.

In the case of park drives and roadways not subjected to heavy, constant traffic, and where a more grainy and coarse surface is desired, the surface finish specified above may be omitted and the following method of finishing adopted:

As the bituminous concrete is raked to grade, and just before the roller comes on it, spread dry stone chips or coarse torpedo sand, evenly with swinging motions of a shovel, until the surface is barely covered. Then roll thoroughly as specified in the preceding paragraph relating to method of laying. If bare spots appear under the roller, sprinkle more chips or sand and continue the rolling until the whole surface is fairly covered.

After the sand or stone chips have worn into the surface, the street shall be swept, all excess of surfacing material removed and the street left clean.

Asphaltic Cement and Flux.—Use the specifications for asphalt, asphalt cement and flux reported by the sub-committee on sheet asphalt pavement specifications and adopted by the Society.

Coal Tar Cement.—The coal tar cement shall be residue of the distillation of coal tar only, and shall be refined for the special purpose of making a paving cement.

No mixture of hard pitch with the lighter oils of coal tar will be permitted.

Its specific gravity shall be not less than 1.20 nor more than 1.29 at 69°F.

The melting-point determined by the cube method shall be not less than 100°F., and not more than 115°F.

It shall contain not less than 15 per cent., nor more than 30 per cent. of free carbon insoluble in benzol.

It shall be free from water as determined by distillation and shall show upon ignition not more than ½ per cent. of inorganic matter.

No distillate shall be obtained lower than 338°F., and up to 600° not less than 5 per cent. and not more than 20 per cent. of distillate shall be obtained. The distillate shall be of a gravity of not less than 1.03 at 60°F. The residue shall have a melting-point of not more than 165°F. In making this distillation an 8-ounce glass retort shall be used and the thermometer suspended so that before applying the heat the bulb of the thermometer is ½ in. above the surface of the liquid. The melting-point of the pitch shall be determined by suspending a ½-in. cube in a beaker of water 1 in. above the bottom. The temperature shall be raised 9° per minute from 60°F. The temperature recorded the instant the pitch touches the bottom shall be considered the melting-point of the pitch. In testing the original materials the initial temperature shall be 40°F.

WATER-GAS TAR CEMENT

1. The specific gravity at 25°C. shall be between 1.155 and 1.170.

2. On extraction with cold carbon disulphide at room temperature for 20 minutes, not less than 97½ per cent. shall be soluble.

3. When tested in a penetrometer at 25°C. with a No. 2 needle under 100 grams load for 5 seconds, it shall have a penetration of not less than 27.5 mm. and not more than 32.5 mm.

4. When 100 c.c. are distilled in a 250-c.c. Engler flask according to the method proposed by the American Society for Testing Materials, the loss by weight shall be within the following limits:

From start to 170°C.	0
170 to 225°C.	not over ½ per cent.
225 to 270°C.	from 2 to 6 per cent.
270 to 300°C.	from 5 to 9 per cent.
Residue	not less than 84 per cent.

SPECIFICATIONS FOR BITULITHIC PAVEMENT ON ANY APPROVED FORM OF FOUNDATION

Wearing Surface.—On the foundation prepared as herein above specified, shall be laid the Bitulithic Wearing Surface and Seal Coat, described below, so as to have a thickness of two (2) inches after thorough compression. The Wearing Surface shall be composed of hard crushed stone, sand, and Bitulithic Cement.

The Bitulithic Cement herein specified besides being produced under the direction, processes, supervision and laboratory inspection of, and with ingredients approved by Warren Brothers Company, shall in all respects comply with the specifications for Asphalt Cement contained in the Sheet Asphalt Specifications of the American Society of Municipal Improvements.

Either of the two following methods and apparatus shall be used in the preparation of the wearing surface.

1. The stone and sand shall be heated in a rotary dryer and while still hot separated into the desired number of different sizes by means of a rotary screen having a minimum screen opening of about $\frac{1}{10}$ of an inch and a maximum opening of about $1\frac{1}{2}$ inch. The openings in the successive screen sections up to one-half inch ($\frac{1}{2}$ in.) size, shall not vary more than one-fourth of an inch ($\frac{1}{4}$ in.) and not more than three-quarters of an inch ($\frac{3}{4}$ in.) for the sizes larger than one-half inch ($\frac{1}{2}$ in.). The aggregate thus separated shall pass into a bin having sections or compartments corresponding to the screen sections. From these compartments the aggregate shall pass into a weigh box, resting on a multi-beam scale. The desired amount of aggregates from each of the above compartments, shall be accurately weighed separately on the scale and the batch dropped into a "twin pug" mixer, where it shall be intimately associated and thoroughly commingled with a predetermined quantity of Bitulithic Cement sufficient to coat all particles of the aggregate and to fill the voids in same.

2. The stone and sand shall first be carefully measured as to sizes and a definite quantity of each size shall then be fed into an elevator terminating in a hopper or bin which discharges into a rotary dryer or heater, both hopper and heater being so designed as to keep each batch by itself until heated. From the rotary heater the batch of mineral aggregate shall pass into a rotary cylindrical mixer containing blades, spirals or other devices for producing a uniform mixture of the mineral aggregate with a predetermined quantity of the Bitulithic Cement sufficient to coat all the particles of the aggregate and to fill the voids in same.

The different sized particles of stone and sand ranging in size from impalpable powder to about one-half the thickness of the wearing surface, shall be combined in such proportions as to secure in the mineral aggregate density, or low percentage of voids, and inherent stability or resistance to displacement, producing an aggregate which when combined with the Bitulithic Cement and laid in place and compacted will form a street paving structure consisting of mineral aggregate of different sizes and the Bitulithic Cement which permeates the entire mass, fills the voids and unites the various particles thereof. If the crushed stone and sand do not contain enough finely divided particles or impalpable powder to produce a low percentage of voids in the aggregate, the deficiency shall be made up by the addition of any other suitable fine mineral matter.

The mixture and ingredients thereof shall be maintained at a temperature consistent with good workmanship. The mixture when reaching the street shall be hot enough to allow of being easily spread and raked and shall not be so hot as to injure the Bitulithic Cement.

Surface Finish or Seal Coat.—There shall be spread over the Bitulithic surface mixture a seal coat, using per square yard of Bitulithic pavement approximately one-fourth ($\frac{1}{4}$) gallon of Bitulithic Cement, into which shall be incorporated approximately twenty-five (25) pounds of mineral aggregate not larger than one-quarter ($\frac{1}{4}$) inch diameter. After spreading the seal coat, it shall be thoroughly rolled into the Bitulithic surface mixture. On grades a coarser aggregate may be used.

General.—Each layer of the work shall be kept as free as possible from dirt, so that it will unite with the succeeding layer.

The bituminous composition or cement shall in each case be free from water and shall be especially refined to remove volatile and other matter susceptible to atmospheric influences.

Warren Brothers Company, owner of the patents used in the construction of the Bitulithic Pavement, shall file with the proper official or board which is about to receive bids for the work, a properly executed binding agreement to furnish any contractor desiring to bid for the work all the necessary Bitulithic Surface material, mixed ready for use, and Bitulithic Cement, and the sand, gravel, or stone screenings for the surface finish course, in accordance with Sections "Wearing Surface" and "Surface Finish," at a definite reasonable price per square yard. Said price shall include a license to use all of the patents required in the construction of the Bitulithic Pavement as herein specified.

The acceptance of bids by.....
and the letting of a contract for the same shall be deemed by Warren Brothers Company to be an acceptance of its proposal by .....
..... and by the Contractor to whom such contract shall be awarded, and are all that shall be necessary to bind Warren Brothers Company to said agreement. The filing of a bid under these specifications will be construed as an acceptance of the terms of the license agreement filed by the Warren Brothers Company, at the price fixed in said agreement, which is on file with the proper official or board.

Boston, Mass., Nov. 27th, 1915.

Mr. Chas. C. Brown, C. E., Secretary,
American Society of Municipal Improvements,
Indianapolis, Indiana.

Dear Sir:

In accordance with our verbal statement to the Sub-Committee on Bituminous Paving Specifications, the General Committee on Paving Specifications, and subsequently on the floor of the Convention of the Society at Dayton, we hereby agree that a pavement constructed by the use of the proportions of mineral aggregate enumerated below, combined with bituminous cement, will not possess the inherent stability and density covered by the claims of and, therefore, will not infringe on our patents. The proportions referred to as adopted by the Society at Dayton convention being as follows:

Bitumen	7 to 9 per cent.
Passing 200 mesh screen	7 to 10 per cent.
Passing 80 mesh screen	10 to 20 per cent.
Passing 40 mesh screen	10 to 25 per cent.
Passing 20 mesh screen	10 to 25 per cent.
Passing 8 mesh screen	10 to 20 per cent.
Passing 4 mesh screen	15 to 20 per cent.
Passing 2 mesh screen	5 to 10 per cent.

Very truly yours,
WARREN BROTHERS COMPANY,
By (Signed) Geo. C. Warren,
President.

Specifications for Broken Stone and Gravel Roads, Adopted October 12, 1916, by the American Society of Municipal Improvements

SPECIFICATIONS FOR BROKEN STONE ROAD

1. General Description.—The broken stone road shall consist of three courses of broken stone, separately constructed, laid to conform to the required grades and cross-sections and constructed as hereinafter specified.

BROKEN STONE

2. Quality of Broken Stone.—All broken stone shall be clean, rough surfaced and sharp angled, of compact texture and uniform grain.

Tests for Broken Stone.—The broken stone shall be subjected to abrasion tests and toughness tests conducted by the engineer in accordance with methods adopted by the American Society for Testing Materials, August 15, 1908. It shall show a "French coefficient of wear" of not less than 7.0 and its toughness shall be not less than 6.0.

3. Sizes.—The product of the crusher shall be passed over a rotary screen with sections having respectively circular openings of the following dimensions: First section, five-eighths (⅝) inch holes; second section, one and one-quarter 1¼)(inch holes; third section, two and one-quarter (2¼) inch holes; fourth section, three and one-half (3½)inch holes. If so directed the first section of the screen shall be fitted with a dust jacket having one-quarter (¼) inch openings so placed as to separate the dust from the product passing through the first section. The screening plant shall also be fitted with a tailing chute so that no stone

failing to pass the largest openings will fall into the bin for No. 4 size broken stone. The various sizes of broken stone shall be caught in separate bins and shall be designated as follows:

Dust, all passing through one-quarter (¼)-inch screen.

Screenings all passing through five-eighths (⅝)-inch screen.

No. 1 size, passing through five-eighths (⅝)-inch screen and over one-quarter (¼)-inch screen.

No. 2 size, passing over five-eighths (⅝)-inch screen and through one and one-quarter (1¼)-inch screen.

No. 3 size, passing over one and one-quarter (1¼)-inch screen and through two and one-quarter (2¼)-inch screen.

No. 4 size, passing over two and one-quarter (2¼)-inch screen and through three and one-half (3½)-inch screen.

Tailings, passing over three and one-half (3½)-inch screen.

Portable Plants.—Portable crushing and screening plants shall be operated as directed.

Stationary Plants.—If broken stone is to be supplied from stationary crushing and screening plants, the several sizes of broken stone shall not be used unless samples have been previously approved by the engineer. The various sizes of broken stone furnished shall be substantially the same as the samples approved.

CONSTRUCTION

4. First Course.—After the sub-grade or sub-base course shall have been prepared as specified, a course of No. 4 broken stone shall be evenly spread so that it shall have after rolling the required thickness of three and one-half (3½) inches. The depth of loose broken stone shall be gaged by the use of strings between iron stakes, as directed. The spreading of the broken stone must be from piles dumped on boards provided for the purpose or from piles dumped alongside the road, or as directed by the engineer. This course shall be thoroughly rolled with a ten (10) to fifteen (15) ton road roller. The rolling shall begin at the sides of the road and continue towards the center and shall be kept up until there is no disturbance of the stone ahead of the roller. After the completion of the rolling, no teaming other than that necessary for bringing on the broken stone for the next course shall be allowed over the rolled broken stone. Should it be apparent after the rolling of the first course that the subgrade material shall have become churned up into or mixed with the broken stone of this course, whether by reason of the rolling, or by hauling over the broken stone or otherwise, the contractor shall at his own expense remove and replace such mixture of sub-grade material and broken stone with clean broken stone of the proper size and shall roll the material to produce a uniform, firm and even first course as required.

5. Second Course.—On the completed first course shall be spread, in the manner specified in the preceding paragraph, No. 3 broken stone to form the second course. This broken stone shall be evenly spread to such a depth that it shall have after rolling the required thickness of two and one-half (2½) inches. After the second course shall be compacted under the same provisions as prescribed for the first course, it shall be evenly covered with a thin layer of screenings. The quantity of screenings to be used shall be just sufficient to cover the larger stones and care shall be exercised to avoid the use of an excess of the screenings. This covering shall then be rolled as heretofore provided. When the rolling shall have been completed the surface of the second course shall be firm, even and true to the lines, grades and cross-sections.

6. Third Course.—On the completed second course shall be spread in the manner above specified for the first course No. 3 broken stone to form the third course. This broken stone shall be evenly spread to such a depth that it will have after rolling the required thickness of two and one-half (2½) inches. After the third course shall have been compacted under the same provisions as prescribed for the first course, it shall be evenly covered with a thin layer of screenings. The quantity of screenings to be used shall be just sufficient to cover the larger stones and care shall be exercised to avoid the use of an excess of the screenings. This covering shall then be rolled as heretofore provided except that water shall be used in connection with the rolling as follows: After the screenings shall have been lightly rolled, water shall be sprinkled on the road surface just ahead of the roller in such quantity as will prevent the sticking to the wheels of the roller of the fine material on the surface, and the combined spreading of screenings, watering and rolling shall be continued until the

voids of the broken stone become so filled with the finer particles as to result in a wave of grout being pushed along the road surface by the front wheel of the roller. When the rolling shall have been completed the surface of the third course shall be firm, even and true to the lines, grades and cross-sections. After the third course has been compacted, puddled and filled as above specified, it shall be evenly covered with a thin layer of screenings. Should at any time, after its construction and prior to the acceptance of the road, the larger stone be visible in the surface of the road, the contractor shall, without extra allowance, spread, sprinkle and roll sufficient screenings to completely cover the same.

PAYMENT

7. Measurement and Payment.—The quantity of broken stone road to be paid for shall be the number of square yards, measured horizontally, satisfactorily completed in accordance with specifications. The price stipulated shall include the furnishing, crushing and screening of the different sizes of broken stone, the placing, rolling and watering of the broken stone, and all work and expenses incidental to the completion of the broken stone road.

SPECIFICATIONS FOR BROKEN STONE ROAD WITH BITUMINOUS SURFACE

BROKEN STONE ROAD

8. General Description.—The broken stone road shall consist of three courses of broken stone, separately constructed, laid to conform to the required grades and cross-sections and constructed as hereinafter specified.

BROKEN STONE

9. Quality of Broken Stone.—All broken stone shall be clean, rough surfaced and sharp angled, of compact texture and uniform grain.

Tests for Broken Stone.—The broken stone shall be subjected to abrasion tests and toughness tests conducted by the engineer in accordance with methods adopted by the American Society for Testing Materials, August 15, 1908. The broken stone used for the construction of the first and second courses shall show a French coefficient of wear of not less than 7.0 and its toughness shall be not less than 6.0. The broken stone used for the construction of the third course and in connection with the bituminous surface shall show a French coefficient of wear of not less than 11.0 and its toughness shall not be less than 13.0.

10. Sizes.—The sizes shall be in accordance with the requirements as stated in the paragraph entitled "Sizes" in the specifications for "Broken Stone Road."

CONSTRUCTION

11. First Course.—After the sub-grade or sub-base course shall have been prepared as specified, a course of No. 4 broken stone shall be evenly spread so that it shall have, after rolling, the required thickness of three and one-half ($3\frac{1}{2}$) inches. The depth of loose broken stone shall be gaged by the use of strings between iron stakes as directed. The spreading of the broken stone must be from piles dumped on boards provided for the purpose or from piles dumped alongside the road, or as directed by the engineer. This course shall be thoroughly rolled with a twelve (12) to fifteen (15)-ton road roller. The initial rolling shall begin at the sides of the road and continue towards the center and shall be kept up until the stone is keyed together and there is no disturbance of the stone ahead of the roller. After the first course has been compacted, it shall be evenly covered with a thin layer of screenings. The quantity of screenings to be used shall be just sufficient to cover the larger stones and care shall be exercised to avoid the use of an excess of the screenings. The covering shall then be rolled as heretofore provided except that water shall be used in connection with the rolling as follows: After the screenings shall have been lightly rolled, water shall be sprinkled on the road surface just ahead of the roller in such quantity as will prevent the sticking to the wheels of the roller of the fine material on the surface, and the combined spreading of screenings, watering and rolling shall be continued until the voids of the broken stone become so filled with the finer particles as to result in a wave of grout being pushed along the road surface by the front wheel of the roller. After the completion of the rolling, no teaming other than that necessary for bringing on the broken stone for the next course shall be allowed over the rolled broken stone. Should it be apparent after the

rolling of the first course that the subgrade material shall have become churned up into or mixed with the broken stone of this course, whether by reason of the rolling, or by hauling over the broken stone or otherwise, the Contractor shall at his own expense remove and replace such mixture of sub-grade material and broken stone with clean broken stone of the proper size and shall roll the material to produce a uniform, firm and even first course as required.

12. Second Course.—On the completed first course shall be spread, in the manner specified in the preceding paragraph, No. 4 broken stone to form the second course. This broken stone shall be evenly spread to such a depth that it shall have, after rolling, the required thickness of three and one-half (3½) inches. The second course shall be compacted, puddled with screenings and water, and finished under the same provisions as prescribed for the first course. When the rolling shall have been completed, the surface of the second course shall be firm, even and true to the lines, grades and cross-sections. If the surface is not slightly rough, so as to afford a sufficient mechanical bond for the third course, it shall be broomed.

13. Third Course.—On the completed second course shall be spread, in the manner above specified for the first course, No. 3 broken stone to form the third course. This broken stone shall be evenly spread to such a depth that it will have, after rolling, the required thickness of two and one-half (2½) inches. The third course shall be compacted and puddled with screenings and water under the same provisions as prescribed for the second course, and when the rolling shall have been completed, the surface of the third course shall be firm, even and true to the lines, grades and cross-sections. After the third course has been compacted, puddled and filled as above specified, it shall be evenly covered with a thin layer of screenings. Should at any time, after the construction of the third course and prior to the application of bituminous material thereon, the larger stone be visible in the surface of the road, the contractor shall, without extra allowance, spread, sprinkle and roll sufficient screenings to completely cover the same. Each section of the broken stone road shall be subjected to traffic for at least one month before the construction of the bituminous surface thereon.

BITUMINOUS SURFACE

14. Description Bituminous Surface.—The bituminous surface shall consist of one application of refined tar covered with a layer of No. 1 broken stone constructed as hereinafter specified.

BITUMINOUS MATERIAL

15. Refined Tar.—Refined tar used in the construction of the bituminous surface shall conform with either one of the specifications covering the chemical and physical properties of refined tars included under the item entitled "Refined Tars for Surface Treatments."

16. Heating Refined Tar.—The refined tar shall be heated in kettles or tanks so designed as to admit of even heating of the entire mass, with an efficient and positive control of the heat at all times. It shall be heated as directed by the engineer to a temperature between 93°C. (200°F.) and 121°C. (250°F.). All refined tar heated beyond 121°C. (250°F.) shall be rejected. No tar shall be heated in kettles or tanks containing any oil or asphalt cement. Before changing from one type to another, kettles or tanks shall be scrupulously cleaned in order to avoid mixtures of the two. Any mixtures of different kinds of bituminous materials shall be rejected.

Thermometers Furnished by Contractors.—The contractor shall provide a sufficient number of accurate, efficient, stationary thermometers for determining the temperature of the refined tar in kettles or tanks.

CONSTRUCTION

17. Preparation of Surface of Road.—Prior to the application of the refined tar, the surface of the broken stone road, when thoroughly dry, shall be swept clean of all dust, dirt or other loose material with horse or power driven brooms and bass or other fine fiber brooms, or with stiff fiber band brooms and bass or other fine fiber brooms, as directed by the engineer. When the cleaning is completed the upper surface of the No. 3 broken stone shall be exposed, forming a clean mosaic surface.

18. Application of Refined Tar.—After the surface shall have been cleaned to the satisfaction of the engineer, and when thoroughly dry, the refined tar shall be uniformly applied over the prepared surface of the road by means of a pressure distributer as hereinafter

specified and in accordance with the directions of the engineer. The refined tar, when applied, shall have a temperature between 93°C. (200°F.) and 121°C. (250°F.). The total amount of refined tar to be used in the construction of the bituminous surface shall be applied in one application and shall not be less than one-quarter (¼) nor more than one-half (½) gallon per square yard, the precise quantity being determined by the engineer.

Pressure Distributer.—The pressure distributer employed shall be so designed and operated as to distribute the refined tar specified uniformly under a pressure of not less than twenty (20) pounds nor more than seventy-five (75) pounds per square inch in the amount and between the limits of temperature specified. It shall be supplied with an accurate stationary thermometer in the tank containing the refined tar and with an accurate pressure gage so located as to be easily observed by the engineer while walking beside the distributer. It shall be so operated that, at the termination of each run, the refined tar will be at once shut off. It shall be so designed that the normal width of application shall be not less than six (6) feet and so that it will be possible on either side of the machine to apply widths of not more than two (2) feet. The distributer shall be provided with tires of widths dependent upon the following relationship between the pressure per square inch of tire and the diameter of the wheel; for a two (2)-foot diameter wheel, five hundred (500) pounds shall be the maximum pressure per linear inch of width per wheel, an additional pressure of thirty (30) pounds per inch being allowed for each additional three (3) inches in diameter.

19. Application of No. 1 Broken Stone.—Immediately after the application of the refined tar, a layer of dry No. 1 broken stone, not to exceed three-eighths (⅜) of an inch in thickness, shall be spread and broomed as directed by the engineer over the surface of the refined tar and shall be at once rolled as directed by the engineer with a roller weighing between eight (8) and fifteen (15) tons.

20. Seasonal and Weather Limitations.—No refined tar shall be applied when the air temperature in the shade is below 10°C. (50°F.), except by the written permission of the engineer.

PAYMENT

21. Measurement and Payment.—The quantity of broken stone road with bituminous surface to be paid for under this item shall be the number of square yards, measured horizontally, satisfactorily completed in accordance with the specifications. The price stipulated in this item shall include the furnishing, crushing and screening of the different sizes of broken stone, the placing, rolling and watering of the broken stone, the heating and distributing of the refined tar, and all materials, work and expenses incidental to the completion of the broken stone road with bituminous surface except the furnishing of the refined tar, which will be included for payment under the item entitled "Refined Tars for Surface Treatments."

SPECIFICATIONS FOR REFINED TARS FOR SURFACE TREATMENTS

22. Previous Service.—The contractor will be required to show to the satisfaction of the engineer, that the company manufacturing the refined tar he proposes to use under a given specification has, for a period of at least two years, manufactured refined tar in a thoroughly equipped plant; and that refined tar manufactured of bituminous material obtained from a similar source to that which he proposes to use shall have been in continuous and successful use in the surface treatment of broken stone roads for a period of at least two years previous to the date of the letting in which his proposal was submitted.

23. Refined Tar "A" Optional With Refined Tar "B."

(I) Refined tar "A" shall be homogeneous, free from water and shall not foam when heated to 121°C. (250°F.).

(II) Its specific gravity at a temperature of 25°C. (77°F.) shall be not less than 1.140 nor more than 1.180.

(III) When tested by means of the New York Testing Laboratory Float Apparatus, the float shall not sink in water maintained at 50°C. (122°F.) in less than 50 seconds nor more than 110 seconds.

(IV) Its bitumen as determined by its solubility in chemically pure carbon disulphide at room temperature, shall be not less than 95.0 per cent. and it shall show not more than 0.2 per cent. ash upon ignition of the material insoluble in carbon disulphide.

(V) When distilled according to the tentative method recommended by Committee D-4 of the American Society for Testing Materials in 1911, it shall yield no distillate at a temperature lower than 170°C. (338°F.); not more than 20.0 per cent. shall distill below 270°C. (518°F.), and not more than 30.0 per cent. shall distill below 300°C. (572°F.).

(V) The melting-point, as determined in water by the cube method, of the pitch residue remaining after distillation to 300°C. (572°F.) in accordance with the test described in Clause (V) shall be not more than 75°C. (167°F.).

24. Refined Tar "B" Optional With Refined Tar "A."

(I) Refined tar "B" shall be homogeneous, free from water, and shall not foam when heated to 121°C. (250°F.).

(II) Its specific gravity at a temperature of 25°C. (77°F.) shall be not less than 1.170 nor more than 1.220.

(III) When tested by means of the New York Testing Laboratory Float Apparatus, the float shall not sink in water maintained at 50°C. (122°F.) in less than 40 seconds nor more than 100 seconds.

(IV) Its bitumen as determined by its solubility in chemically pure carbon disulphide at room temperature, shall be not less than 85.0 per cent. nor more than 95.0 per cent., and it shall show not more than 0.2 per cent. ash upon ignition of the material insoluble in carbon disulphide.

(V) When distilled according to the tentative method recommended by Committee D-4 of the American Society for Testing Materials in 1911, it shall yield no distillate at a temperature lower than 170°C. (338°F.); not more than 20.0 per cent. shall distill below 270°C. (518°F.), and not more than 25.0 per cent. shall distill below 300°C. (572°F.).

(VI) The melting-point, as determined in water by the cube method, of the pitch residue remaining after distillation to 300°C. (572°F.) in accordance with the test described in Clause (V) shall be not more than 75°C. (167°F.).

25. Delivery.—The refined tar shall be delivered in suitable containers, far enough in advance of its use in the work to permit the necessary tests to be made. Each container shall be plainly labeled with the trade name of the refined tar, name of manufacturer, gross weight and net weight. Each shipment and each carload shall be kept separate.

Bills of Lading.—The contractor shall furnish the engineer on or before the arrival of each shipment at or near the site of the work, bills of lading, or correct copies thereof, which shall state the trade name of the refined tar, and the name and address of the company manufacturing and supplying it.

Samples.—Samples will be taken by the engineer from each carload of refined tar when delivered at the work, unless satisfactory arrangements can be made for sampling before shipment. Such samples shall be analyzed by the engineer to assure the delivery of a refined tar of the specified quality and to determine, for purpose of payment, the quantity of bitumen.

26. Work Included.—Under this item the contractor shall furnish and deliver on the work at such points as directed refined tar which conforms with the specifications of either refined tar "A" or refined tar "B."

27. Measurement and Payment.—The quantity of bitumen in the refined tar, to be paid for under this item, shall be the number of tons, determined in accordance with the paragraph headed "Samples" contained in the refined tar placed on the road in accordance with the specifications and requirements, or used as directed for other purposes. The percentage of bitumen determined by an average of the analysis of the acceptable samples taken by the engineer during a given month shall be used as the basis for payment for the refined tar used during that month. Refined tar that is wasted shall not be included in the measurement under this item. The price stipulated in this item shall include the cost of furnishing, hauling and delivering the refined tar at the work, and all expenses incidental thereto.

SPECIFICATIONS FOR BITUMINOUS MACADAM PAVEMENT

28. General Description.—The bituminous macadam pavement shall consist of three courses of broken stone, separately constructed, laid to conform to the required grades and cross-sections, and constructed as hereinafter specified with bituminous material incorporated with the top or third course.

BROKEN STONE

29. Quality of Broken Stone.—All broken stone shall be clean, rough surfaced and sharp angled, of compact texture and uniform grain.

Tests for Broken Stone.—The broken stone shall be subjected to abrasion tests and toughness tests conducted by the engineer in accordance with methods adopted by the American

Society for Testing Materials, August 15, 1908. The broken stone used for the construction of the first and second courses shall show a French coefficient of wear of not less than 7.0 and its toughness shall be not less than 6.0. The broken stone used for the construction of the third course and for the first and second applications of No. 1 broken stone shall show a French coefficient of wear of not less than 11.0 and its toughness shall not be less than 13.0.

30. Sizes.—The sizes shall be in accordance with requirements as stated in the paragraph entitled "Sizes" in the specifications for "Broken Stone Road."

CONSTRUCTION

31. First Course.—After the sub-grade or sub-base course shall have been prepared as specified, a course of No. 4 broken stone shall be evenly spread so that it shall have, after rolling, the required thickness of three and one-half (3½) inches. The depth of loose broken stone shall be gaged by the use of strings between iron stakes, as directed. The spreading of the broken stone must be from piles dumped on boards provided for the purpose or from piles dumped alongside the road, or as directed by the engineer. This course shall be thoroughly rolled with a twelve (12) to fifteen (15)-ton road roller. The initial rolling shall begin at the sides of the road and continue towards the center and shall be kept up until the stone is keyed together and there is no disturbance of the stone ahead of the roller. After the first course has been compacted, it shall be evenly covered with a thin layer of screenings. The quantity of screenings to be used shall be just sufficient to cover the larger stones and care shall be exercised to avoid the use of an excess of the screenings. This covering shall then be rolled as heretofore provided except that water shall be used in connection with the rolling as follows: After the screenings shall have been lightly rolled, water shall be sprinkled on the road surface just ahead of the roller, in such quantity as will prevent the sticking to the wheels of the roller of the fine material on the surface, and the combined spreading of screenings, watering and rolling shall be continued until the voids of the broken stone become so filled with the finer particles as to result in a wave of grout being pushed along the road surface by the front wheel of the roller. After the completion of the rolling, no teaming other than that necessary for bringing on the broken stone for the next course shall be allowed over the rolled broken stone. Should it be apparent after the rolling of the first course that the sub-grade material shall have become churned up into or mixed with the broken stone of this course, whether by reason of the rolling, or by hauling over the broken stone or otherwise, the contractor shall at his own expense remove and replace such mixture of sub-grade material and broken stone with clean broken stone of the proper size and shall roll the material to produce a uniform, firm and even first course as required.

32. Second Course.—On the completed first course shall be spread, in the manner specified in the preceding paragraph, No. 4 broken stone to form the second course. This broken stone shall be evenly spread to such a depth that it shall have, after rolling, the required thickness of three and one-half (3½) inches. The second course shall be compacted, puddled with screenings and water, and finished under the same provisions as prescribed for the first course. When the rolling shall have been completed, the surface of the second course shall be firm, even and true to the lines, grades and cross-sections. If the surface is not slightly rough, so as to afford a sufficient mechanical bond for the third course, it shall be broomed.

TOP COURSE, BITUMINOUS MACADAM PAVEMENT

33. Description of Top Course.—The top course of the bituminous macadam pavement shall consist of a third course of broken stone and two applications of bituminous material, each application being followed by the distribution of a layer of No. 1 broken stone, constructed as hereinafter specified.

34. Asphalt Cement and Refined Tar.—The asphalt cement or refined tar, hereinafter referred to as bituminous material, used in the construction of the third course of the bituminous macadam pavement shall conform with either one of the specifications covering the chemical and physical properties of bituminous materials included under the item entitled "Asphalt Cements and Refined Tars for Bituminous Macadam Pavement."

35. Heating Bituminous Materials.—Bituminous materials shall be heated in kettles or tanks so designed as to admit of even heating of the entire mass, with an efficient and positive control of the heat at all times. Asphalt cement shall be heated as directed by

the engineer to a temperature between 135°C. (275°F.) and 177°C. (350°F.). All asphalt cement heated beyond 177°C. (350°F.) shall be rejected. Refined tar shall be heated as directed by the engineer to a temperature between 93°C. (200°F.) and 121°C. (250°F.). All refined tar heated beyond 121°C. (250°F.) shall be rejected. No tar shall be heated in kettles or tanks containing any oil or asphalt cement. Before changing from one type of material to another, kettles or tanks shall be scrupulously cleaned in order to avoid mixtures of the two. Any mixtures of different kinds of bituminous materials shall be rejected.

Thermometers Furnished by Contractors.—The contractor shall provide a sufficient number of accurate, efficient, stationary thermometers for determining the temperature of the bituminous material in kettles or tanks.

CONSTRUCTION

36. Third Course of Broken Stone.—On the completed second course, when thoroughly dry, shall be spread, in the manner above specified for the first course, dry No. 3 broken stone to form the third course. This broken stone shall be evenly spread to such a depth that it will have, after rolling, the required thickness of two and one-half (2½) inches. The third course shall be thoroughly compacted by dry rolling until the fragments of broken stone have just keyed together in accordance with the same provisions covering rolling as prescribed for the "First Course."

37. First Application of Bituminous Material.—After the third course of broken stone shall have been thoroughly compacted as specified and when clean and thoroughly dry, the bituminous material shall be uniformly applied over the prepared surface of the third course by means of a pressure distributer as hereinafter specified. The asphalt cement, when applied, shall have a temperature between 135°C. (275°F.) and 177°C. (350°F.). The refined tar, when applied, shall have a temperature between 93°C. (200°F.) and 121°C. (250°F.). The total amount of bituminous material to be used in the first application shall not be less than one and one-half (1½) gallons nor more than one and three-quarters (1¾) gallons per square yard, the precise quantity being determined by the engineer.

Pressure Distributer.—The pressure distributer employed shall be so designated and operated as to distribute the bituminous materials specified uniformly under a pressure of not less than twenty (20) pounds nor more than seventy-five (75) pounds per square inch in the amount and between the limits of temperature specified. It shall be supplied with an accurate stationary thermometer in the tank containing the bituminous material and with an accurate pressure gage so located as to be easily observed by the engineer while walking beside the distributer. It shall be so operated that, at the termination of each run, the bituminous material will be at once shut off. It shall be so designed that the normal width of application shall be not less than six (6) feet and so that it will be possible on either side of the machine to apply widths of not more than two (2) feet. The distributer shall be provided with wheels having tires each of which shall not be less than eighteen (18) inches in width, the allowed maximum pressure per square inch of tire being dependent upon the following relationship between the aforesaid pressure and the diameter of the wheel: For a two (2)-foot diameter wheel, two hundred and fifty (250) pounds shall be the maximum pressure per linear inch of width of tire per wheel, an additional pressure of twenty (20) pounds per inch being allowed for each additional three (3) inches in diameter.

38. First Application of No. 1 Broken Stone.—Immediately after the application of the bituminous material, a layer of dry No. 1 broken stone, not to exceed three-eighths (⅜) of an inch in thickness, shall be spread as directed by the engineer over the surface of the bituminous material and shall be at once rolled as directed by the engineer with a roller weighing between twelve (12) and fifteen (15) tons. During the rolling process, additional No. 1 broken stone shall be applied and broomed until the voids in the upper portion of the third course are filled to the satisfaction of the engineer.

39. Second Application of Bituminous Material.—Prior to the second application of bituminous material, all loose No. 1 broken stone shall be swept from the surface of the pavement. When thoroughly clean and dry, a second application of bituminous material shall be uniformly applied over the surface by means of a pressure distributer as specified above. When applied the asphalt cement shall have a temperature between 135°C. (275°F.) and 177°C. (350°F.). When applied the refined tar shall have a temperature between 93°C. (200°F.) and 121°C. (250°F.). The total amount of bituminous material to be used in the second application shall not be less than one-half (½) gallon nor more than three-quarters (¾) gallon per square yard, the precise quantity being determined by the engineer.

40. Second Application of No. 1 Broken Stone.—Immediately after the second application of bituminous material, a layer of dry No. 1 broken stone, not to exceed three-eighths (⅜) of an inch in thickness, shall be spread and broomed as directed by the engineer over the surface of the bituminous material and thereafter at once rolled as directed by the engineer with a roller weighing between twelve (12) and fifteen (15) tons. The rolling shall be continued and additional No. 1 broken stone shall be applied until a smooth, uniform surface is produced to the satisfaction of the engineer.

41. Seasonal and Weather Limitations.—No bituminous material shall be applied when the air temperature in the shade is below 10°C. (50°F.), except by the written permission of the engineer.

PAYMENT

42. Measurement and Payment.—The quantity of bituminous macadam pavement to be paid for under this item shall be the number of square yards, measured horizontally, satisfactorily completed in accordance with the specifications. The price stipulated in this item shall include the furnishing, crushing and screening of the different sizes of broken stone, the heating and distributing of the bituminous material, and all materials, work and expenses incidental to the completion of the bituminous macadam pavement except the furnishing of the bituminous material, which will be included for payment under the item entitled "Asphalt Cements and Refined Tars for Bituminous Macadam Pavements."

SPECIFICATIONS FOR ASPHALT CEMENTS AND REFINED TARS FOR BITUMINOUS MACADAM PAVEMENTS

43. Previous Service.—The contractor will be required to show, to the satisfaction of the engineer, that the company manufacturing the asphalt cement or refined tar he proposes to use under a given specification has, for a period of at least two years, manufactured asphalt cement or refined tar in a thoroughly equipped plant, and that asphalt cement or refined tar manufactured of bituminous material obtained from a similar source to that which he proposes to use shall have been in continuous and successful use in bituminous pavements constructed by the mixing method or in bituminous macadam pavements for a period of at least two years previous to the date of the letting in which his proposal was submitted.

44. Asphalt Cement "A" Optional With Asphalt Cements "B," "C," "D," and Refined Tars "E" and "F."

(I) Asphalt Cement "A" shall be homogeneous, free from water and shall not foam when heated to 177°C. (350°F.).

(II) It shall show a flash point of not less than 205°C. (400°F.) when tested in the New York State Board of Health Closed Oil Tester.

(III) Its specific gravity, at a temperature of 25°C. (77°F.) shall be not less than 0.960 nor more than 1.000.

(IV) When tested with a standard No. 2 needle by means of a standard penetrometer, it shall show penetrations within the following limits for the conditions stated, the penetrations being expressed in hundredths of a centimeter: 100-gram load, 5 seconds at 25°C. (77°F.), from 100 to 120; 200-gram load, 1 minute at 4°C. (39°F.), not less than 50.

(V) Its melting-point as determined by the cube method shall be not less than 60°C. (140°F.).

(VI) When 50 grams of the material is maintained at a uniform temperature of 163°C. (325°F.) for 5 hours in an open cylindrical tin dish 5½ centimeters (about 2¼ in.) in diameter, with vertical sides measuring approximately 3½ centimeters (about 1½ in.) in depth, the loss in weight shall not exceed 2.0 per cent. of the original weight of the sample.

The penetration of the residue, when tested as described in Clause (IV) with a standard No. 2 needle under a load of 100 grams, for 5 seconds at 25°C. (77°F.) shall be not less than one-half the penetration of the original material tested under the same condition.

(VII) Its bitumen as determined by its solubility in chemically pure carbon disulphide at room temperature, shall be not less than 99.5 per cent.

(VIII) It shall be soluble in chemically pure carbon tetrachloride at room temperature, to the extent of not less than 99.5 per cent. of its bitumen as determined by Clause (VII).

(IX) It shall be soluble in 86 to 88° Baumé paraffin naphtha, of which at least 85.0 per cent. distills between 35° and 65°C. (95° and 149°F.), to the extent of not less than 75.0 per cent. nor more than 85.0 per cent. of its bitumen as determined by Clause (VII).

(X) It shall yield not less than 8.0 per cent. nor more than 12.0 per cent. of fixed carbon.

45. Asphalt Cement "B" Optional With Asphalt Cements "A," "C," "D," and Refined Tars "E" and "F."

(I) Asphalt cement "B" shall be homogeneous, free from water and shall not foam when heated to 177°C. (350°F.).

(II) It shall show a flash point of not less than 205°C. (400°F.) when tested in the New York State Board of Health Closed Oil Tester.

(III) Its specific gravity, at a temperature of 25°C. (77°F.) shall be not less than 1.000 nor more than 1.030.

(IV) When tested with a standard No. 2 needle by means of a standard penetrometer, it shall show penetrations within the following limits for the conditions stated, the penetrations being expressed in hundredths of a centimeter: 100-gram load, 5 seconds, at 25°C. (77°F.), from 90 to 110; 200-gram load 1 minute, at 4°C. (39°F). not less than 15.

(V) Its melting-point as determined by the cube method shall be not less than 30°C. (86°F.).

(VI) When 50 grams of the material are maintained at a uniform temperature of 163°C. (325°F.) for 5 hours in an open cylindrical tin dish 5½ centimeters (about 2¼ in.) in diameter, with vertical sides measuring approximately 3½ centimeters (about 1½ in.) in depth, the loss in weight shall not exceed 2.0 per cent. of the original weight of the sample.

The penetration of the residue, when tested as described in Clause (IV) with a standard No. 2 needle under a load of 100 grams, for 5 seconds at 25°C. (77°F.) shall be not less than one-half the penetration of the original material tested under the same conditions.

(VII) Its bitumen, as determined by its solubility in chemically pure carbon disulphide at room temperature, shall be not less than 99.5 per cent.

(VIII) It shall be soluble in chemically pure carbon tetrachloride at room temperature, to the extent of not less than 99.5 per cent. of its bitumen as determined by Clause (VII).

(IX) It shall be soluble in 86° to 88°Bé. paraffin naphtha, of which at least 85.0 per cent. distills between 35° and 65°C. (95° and 149°F.), to the extent of not less than 75.0 per cent. nor more than 85.0 per cent. of its bitumen as determined by Clause (VII).

(X) It shall yield not less than 9.0 per cent. nor more than 13.0 per cent. of fixed carbon.

46. Asphalt Cement "C" Optional With Asphalt Cements "A," "B," "D," and Refined Tars "E" and "F."

(I) Asphalt cement "C" shall be homogeneous, free from water and shall not foam when heated to 177°C. (350°F.).

(II) It shall show a flash point of not less than 205°C. (400°F.) when tested in the New York State Board of Health Closed Oil Tester.

(III) Its specific gravity, at a temperature of 25°C. (77°F.) shall be not less than 1.025 nor more than 1.045.

(IV) When tested with a standard No. 2 needle by means of a standard penetrometer, it shall show penetrations within the following limits for the conditions stated, the penetrations being expressed in hundredths of a centimeter: 00-gram load, 5 seconds, at 25°C. (77°F.), from 110 to 130; 200-gram load, 1 minute, at 4°C. (39°F.) not less than 30.

(V) Its melting-point as determined by the cube method shall be not less than 40°C. (104°F.).

(VI) When 50 grams of the material are maintained at a uniform temperature of 163°C. (325°F.) for 5 hours in an open cylindrical tin dish 5½ centimeters (about 2¼ in.) in diameter, with vertical sides measuring approximately 3½ centimeters (about 1½ in.) in depth, the loss in weight shall not exceed 2.0 per cent. of the original weight of the sample.

The penetration of the residue, when tested as described in Clause (IV) with a standard No. 2 needle under a load of 100 grams for 5 seconds at 25°C. (77°F.) shall be not less than one-half the penetration of the original material tested under the same conditions.

(VII) Its bitumen, as determined by its solubility in chemically pure carbon disulphide at room temperature, shall be not less than 99.5 per cent.

(VIII) It shall be soluble in chemically pure carbon tetrachloride at room temperature, to the extent of not less than 99.5 per cent. of its bitumen as determined by Clause (VII).

(IX) It shall be soluble in 86° to 88° Bé. paraffin naphtha, of which at least 85.0 per cent. distills between 35° and 65°C. (95° and 149°F.) to the extent of not less than 70.0 per cent. nor more than 80.0 per cent. of its bitumen as determined by Clause (VII).

(X) It shall yield not less than 12.0 per cent. nor more than 17.0 per cent. of fixed carbon.

47. Asphalt Cement "D" Optional With Asphalt Cements "A," "B," "C," and Refined Tars "E" and "F."

(I) Asphalt cement "D" shall be homogeneous, free from water and shall not foam when heated to 177°C. (350°F).

(II) It shall show a flash point of not less than 163°C. (325°F.) when tested in the New York State Board of Health Closed Oil Tester.

(III) Its specific gravity, at a temperature of 25°C. (77°F.) shall be not less than 1.035 nor more than 1.060.

(IV) When tested with a standard No. 2 needle by means of a standard penetrometer. it shall show penetrations within the following limits for the conditions stated, the penetrations being expressed in hundredths of a centimeter; 100-gram load, 5 seconds, at 25°C. (77°F.), from 130 to 160; 200-gram load, 1 minute at 4°C. (39°F.), not less than 30.

(V) When tested by means of the New York Testing Laboratory Float Apparatus, the float shall not sink in water maintained at 66°C. (150°F.) in less than 120 seconds nor more than 180 seconds.

(VI) When 50 grams of the material are maintained at a uniform temperature of 163°C. (325°F.) for 5 hours in an open cylindrical tin dish 5½ centimeters (about 2¼ in.) in diameter, with vertical sides measuring approximately 3½ centimeters (about 1½ in.) in depth the loss in weight shall not exceed 3.0 per cent. of the original weight of the sample.

The penetration of the residue, when tested as described in Clause (IV) with a standard No. 2 needle under a load of 100 grams, for 5 seconds at 25°C. (77°F.) shall be not less than one-half the penetration of the original material tested under the same conditions.

(VII) Its bitumen as determined by its solubility in chemically pure carbon disulphide at room temperature, shall be not less than 94.0 per cent. nor more than 98.0 per cent.

(VIII) It shall be soluble in chemically pure carbon tetrachloride at room temperature, to the extent of not less than 98.5 per cent. of its bitumen as determined by Clause (VII).

(IX) It shall be soluble in 86° to 88° Bé. paraffin naphtha, of which at least 85.0 per cent. distills between 35° and 65°C. (95° and 149°F.) to the extent of not less than 75.0 per cent. nor more than 85.0 per cent. of its bitumen as determined by Clause (VII).

(X) It shall yield not less than 11.0 per cent. nor more than 14.0 per cent. of fixed carbon.

(XI) Upon ignition it shall yield not less than 1.0 per cent. nor more than 3.0 per cent. of ash.

48. Refined Tar "E" Optional With Asphalt Cements "A," "B," "C," "D," and Refined Tar "F."

(I) Refined tar "E" shall be homogeneous, free from water, and shall not foam when heated to 121°C. (250°F.).

(II) Its specific gravity at a temperature of 25°C. (77°F.) shall be not less than 1.150 nor more than 1.200.

(III) When tested by means of the New York Testing Laboratory Float Apparatus, the float shall not sink in water maintained at 50°C. (122°F.) in less than 120 nor more than 150 seconds.

(IV) Its bitumen as determined by its solubility in chemically pure carbon disulphide at room temperature, shall be not less than 95.0 per cent., and it shall show not more than 0.2 per cent. ash upon ignition of the material insoluble in carbon disulphide.

(V) When distilled according to the tentative method recommended by Committee D-4 of the American Society for Testing Materials in 1911, it shall yield not more than 0.5 per cent. distillate at a temperature lower than 170°C. (338°F.); not more than 12.0 per cent. shall distill below 270°C. (518°F.), and not more than 25.0 per cent. shall distill below 300°C. (572°F.).

(VI) The total distillate from the test made in accordance with Clause (V) shall have a specific gravity at a temperature of 25°C. (77°F.) of not less than 0.980 nor more than 1.020.

(VII) The melting-point, as determined in water by the cube method, of the pitch residue remaining after distillation to 300°C. (572°F.) in accordance with the test described in Clause (V), shall be not more than 75°C. (167°F.).

49. Refined Tar "F" Optional With Asphalt Cements "A," "B," "C," "D," and Refined Tar "E."

(I) Refined tar "F" shall be homogeneous, free from water, and shall not foam when heated to 121°C. (250°F.).

(II) Its specific gravity at a temperature of 25°C. (77°F.) shall be not less than 1.180 nor more than 1.300.

(III) When tested by means of the New York Testing Laboratory Float Apparatus, the float shall not sink in water maintained at 50°C. (122°F.) in less than 150 nor more than 180 seconds.

(IV) Its bitumen as determined by its solubility in chemically pure carbon disulphide at room tempeature, shall be not less than 80.0 per cent. nor more than 95.0 per cent., and it shall show not more than 0.2 per cent. ash upon ignition of the material insoluble in carbon disulphide.

(V) When distilled according to the tentative method recommended by Committee D-4 of the American Society for Testing Materials in 1911, it shall yield not more than 0.5 per cent. distillate at a temperature lower than 170°C. (338°F.); not more than 10.0 per cent. shall distill below 270°C. (518°F.), and not more than 20.0 per cent. shall distill below 300°C. (572°F.).

(VI) The total distillate from the test made in accordance with Clause (V) shall have a specific gravity at a temperature of 25°C. (77°F.) of not less than 1.020.

(VII) The melting-point, as determined in water by the cube method, of the pitch residue remaining after distillation to 300°C. (572°F.) in accordance with the test described in Clause (V), shall be not more than 75°C. (167°F.).

50. Delivery.—The asphalt cement or refined tar shall be delivered in suitable containers, far enough in advance of its use in the work to permit the necessary tests to be made. Each container shall be plainly labeled with the trade name of the asphalt cement or refined tar, name of manufacturer, gross weight and net weight. Each shipment and each carload shall be kept separate.

Bills of Lading.—The contractor shall furnish the engineer on or before the arrival of each shipment at or near the site of the work, bills of lading, or correct copies thereof, which shall state the trade name of the asphalt cement or refined tar, and the name and address of the company manufacturing and supplying it.

51. Samples.—Samples will be taken by the engineer from each carload of asphalt cement or refined tar when delivered at the work, unless satisfactory arrangements can be made for sampling before shipment. Such samples shall be analyzed by the engineer to assure the delivery of an asphalt cement or refined tar of the specified quality and to determine, for purpose of payment, the quantity of bitumen.

52. Work Included.—Under this item the contractor shall furnish and deliver on the work at such points as directed an asphalt cement or refined tar which conforms with the specifications of any one of the asphalt cements or refined tars given above.

53. Measurement and Payment.—The quantity of bitumen in the asphalt cement or refined tar, to be paid for under this item, shall be the number of tons, determined in accordance with the paragraph headed "*Samples*," contained in the asphalt cement or refined tar placed in the pavement in accordance with the specifications and requirements, or used as directed for other purposes. The percentage of bitumen determined by an average of the analyses of the acceptable samples taken by the engineer during a given month shall be used as the basis for payment for the asphalt cement or refined tar used during that month. Asphalt cement or refined tar that is wasted shall not be included in the measurement under this item. The price stipulated in this item shall include the cost of furnishing, hauling and delivering the asphalt cement or refined tar at the work and all expenses incidental thereto.

SPECIFICATIONS FOR A GRAVEL ROAD

1. General Description.—The gravel road shall consist of three courses of mixtures of gravel, sand and clay, separately constructed, laid to conform to the required grades and cross-sections and constructed as hereinafter specified. The transverse slope of the finished surface of the gravel roadway shall be one (1) inch per foot.

2. Subgrade.—The subgrade for the gravel roadway shall consist of the natural earth roadbed prepared and rolled until firm, hard and even, and shall conform to the specified cross-section. If sandy or other soil be encountered which will not compact readily under the roller, a small amount of clay, or other means satisfactory to the engineer, shall be used until a firm, even surface is obtained after rolling. Where the proposed grade allows the use of an old roadway for the subgrade, the roadway shall be shaped and rolled to the specified cross-section and elevations and depressions removed so as to form an even surface before the construction of the first course. The roller used shall be a ten (10) to fifteen (15) ton road roller.

3. Shoulders.—After the roadbed has been graded, shoulders of firm earth or other suitable material eight (8) inches in depth after compaction, shall be constructed on each side of the roadbed at such distances apart as may be required to retain the width of gravel specified. No material which contains weeds, sod, roots or other perishable matter and

which will not compact under the roller shall be placed in the shoulders. The shoulders shall extend to the side ditches or gutters with the same transverse slope as required for the finished roadway surface and shall be thoroughly rolled at the same time as the third or wearing course.

MATERIALS

4. Quality of Gravel.—All gravel shall be hard and tough. Gravel which contains over 10 per cent. of disintegrated stone shall not be used.

5. Sizes.—Two mixtures of gravel, sand and clay shall be used hereinafter designated in these specifications as No. 1 product and No. 2 product.

No. 1 product shall consist of a mixture of gravel, sand and clay, with the proportions of the various sizes as follows: All to pass a one and one-half (1½)-inch screen and to have at least 60 and not more than 75 per cent. retained on a one-quarter (¼)-inch screen; at least 25 and not more than 75 per cent. of the total coarse aggregate (material over one-quarter (¼) inch in size) to be retained on a three-quarters (¾)-inch screen; at least 65 and not more than 85 per cent. of the total fine aggregate (material under one-quarter (¼) inch in size) to be retained on a two hundred (200)-mesh sieve.

No. 2 product shall consist of a mixture of gravel, sand and clay, with the proportions of the various sizes as follows: All to pass a two and one-half (2½)-inch screen and to have at least 60 and not more than 75 per cent. retained on a one-quarter (¼)-inch screen; at least 25 and not more than 75 per cent. of the total coarse aggregate to be retained on a one (1)-inch screen; at least 65 and not more than 85 per cent. of the total fine aggregate to be retained on a two hundred (200)-mesh sieve.

6. Test.—The mixture of gravel, sand and clay of the No. 1 product shall be subjected to a cementation test conducted by the engineer in accordance with the method recommended by the Special Committee on "Materials for Road Construction" of the American Society of Civil Engineers in January, 1916, and as described in the 1915 *Proceedin s*, page 2738, except that the test shall be made on material which will pass a one-quarter (¼)-inch screen. Its coefficient of cementation shall not be less than 50.

CONSTRUCTION

7. First Course.—After the subgrade or sub-base course shall have been prepared as specified, a course of No. 2 product shall be evenly spread so that it shall have, after rolling, the required thickness of three (3) inches. The depth of the No. 2 product loose shall be gaged by the use of strings between iron stakes, as directed. The spreading of the mixture of gravel, sand and clay must be from piles dumped on boards provided for the purpose or from piles dumped alongside the road. This course shall be thoroughly rolled with a ten (10) to fifteen (15)-ton road roller. The initial rolling shall begin at the sides of the road and continue towards the center and shall be kept up until there is no disturbance of the No. 2 product ahead of the roller. After the first course has been compacted water shall be sprinkled on the roadway surface just ahead of the roller in such quantity as shall prevent the sticking to the wheels of the roller of the fine material on the surface, and the combined watering and rolling shall be continued until the voids of the gravel become filled with fine particles and until the roadway surface conforms to the specified cross-section. After the completion of the rolling no teaming other than that necessary for bringing on the No. 2 product for the next course shall be allowed over the rolled surface. The surface of the first course shall be maintained in its finished condition until the second course shall have been spread. Should it be apparent after the rolling of the first course that the subgrade or shoulder material shall have become churned up into or mixed with the material of this course, whether by reason of the rolling or by hauling over the surface, or otherwise, the contractor shall at his own expense remove and replace such mixture of No. 2 product and subgrade or shoulder material with No. 2 product and shall roll the material to produce a uniform, firm and even first course as required.

8. Second Course.—On the completed first course shall be spread, in the manner specified in the preceding paragraph, No. 2 product to form the second course. This mixture of gravel, sand and clay shall be evenly spread to such depth that it shall have, after rolling, the required thickness of three (3) inches. The second course shall be compacted, puddled with water and finished under the same provisions as prescribed for the first course. When the rolling shall have been completed, the surface of the second course shall be firm, even and true to the lines, grades and cross-sections.

9. Third Course.—On the completed second course shall be spread, in the manner above specified for the first course, No. 1 product to form the third course. This mixture of gravel, sand and clay shall be evenly spread to such a depth that it will have, after rolling, the required thickness of two (2) inches. The third course shall be compacted and puddled with water under the same provisions as prescribed for the first course. When the rolling shall have been completed, the surface of the third course shall be firm, even and true to the lines, grades and cross-sections. If necessary to satisfactorily bond the roadway surface, the third course shall then be evenly covered with a thin layer of sand or sand and clay and rolled.

PAYMENT

10. Measurement and Payment.—The quantity of gravel road to be paid for shall be the number of square yards, measured horizontally, satisfactorily completed in accordance with the specifications. The price stipulated shall include shaping and rolling of the subgrade, the furnishing of the different products of gravel, sand and clay, the placing, rolling and watering of the several courses, and all work and expenses incidental to the completion of the gravel road. (The quantity of shoulders shall be paid for under a separate item.)

SPECIFICATIONS FOR A BITUMINOUS CONCRETE PAVEMENT, MINERAL AGGREGATE COMPOSED OF ONE PRODUCT OF A STONE CRUSHING PLANT

General Description.—The bituminous concrete wearing course shall consist of a compact mixture of broken stone and asphalt cement or refined tar laid to conform to the required grades and cross-sections, covered with a seal coat of asphalt cement and broken stone chips, and constructed as hereinafter specified.

BROKEN STONE

Quality.—All broken stone shall be clean, rough surfaced and sharp angled, of compact texture and uniform grain.

Tests.—The broken stone shall be subjected to abrasion and toughness tests conducted by the engineer in accordance with methods adopted by the American Society for Testing Materials, August 15, 1908. The broken stone used for the construction of the wearing course shall show a loss on abrasion of not more than 3.5 per cent. or a French coefficient of wear of not less than 11.5, and its toughness shall not be less than 13.0.

Broken Stone Chips.—Broken stone chips shall consist of the product of a stone crushing plant obtained by passing the broken stone through a section of a rotary screen having circular openings three-eighths (⅜) or one-half (½) inch in diameter and over a screen having openings of one-eighth (⅛) or one-quarter (¼) inch.

Broken Stone for Mineral Aggregate.—Broken stone for the mineral aggregate of the wearing course shall consist of one product of a stone crushing and screening plant. It shall conform to the following mechanical analysis, using laboratory screens having circular openings: All of the broken stone shall pass a one and one-quarter (1¼)-inch screen; not more than ten (10) per cent. nor less than one (1) per cent. shall be retained upon a one (1)-inch screen; not more than ten (10) per cent. nor less than three (3) per cent. shall pass a one-quarter (¼)-inch screen.

Heating.—Before entering the mixer, the broken stone for the mineral aggregate shall be heated until thoroughly dry to between 66°C. (150°F.) and 121°C. (250°F.), as directed, in revolving driers in which no flame shall be permitted to come in contact with the broken stone and in which the broken stone shall be continuously agitated during the heating.

BITUMINOUS CEMENT

Asphalt Cement and Refined Tar.—The asphalt cement or refined tar used in the construction of the wearing course of the bituminous concrete pavement shall conform with either one of the specifications covering the chemical and physical properties of bituminous cement included under the item entitled "Asphalt Cements and Refined Tars for Wearing Course of Bituminous Concrete Pavement." If asphalt cement is used in the bituminous concrete wearing course, the same asphalt cement shall be used for the seal coat. If refined tar is used in the bituminous concrete wearing course, an asphalt cement shall be

used in the seal coat and shall conform with either one of the specifications covering the physical and chemical properties of asphalt cements included under the item entitled "Asphalt Cements for Seal Coat for Bituminous Concrete Pavement."

Previous Service.—The contractor will be required to show, to the satisfaction of the engineer, that the company manufacturing the asphalt cement or refined tar he proposes to use under a given specification has, for a period of at least two years, manufactured asphalt cement or refined tar in a thoroughly equipped plant, and that asphalt cement or refined tar manufactured of bituminous material obtained from a similar source to that which he proposes to use, shall have been in continuous and successful use for a period of at least two years in bituminous pavements constructed by the mixing method previous to the date of the letting in which his proposal was submitted.

Heating.—The asphalt cement or refined tar shall be heated in kettles so designed as to admit of even heating of the entire mass, with an efficient and positive control of the heat at all times. Asphalt cement shall be heated as directed to a temperature between 135°C. (275°F.) and 177°C. (350°F.). All asphalt cement heated beyond 177°C. (350°F.), either before or during mixing with the broken stone, shall be rejected. Refined tar shall be heated as directed to a temperature between 93°C. (200°F.) and 135°C. (275°F.). All tar heated beyond 135°C. (275°F.), either before or during mixing with broken stone, shall be rejected. No tar shall be heated in kettles containing any asphalt cement and in like manner no asphalt cement shall be heated in kettles containing any tar. Before changing from one type of material to the other, kettles shall be scrupulously cleaned in order to avoid mixtures of the two. Any such mixtures resulting from this cause shall be rejected.

Thermometers Furnished by Contractor.—The contractor shall provide a sufficient number of accurate, efficient, stationary thermometers for determining the temperatures of the asphalt cement or refined tar in kettles.

CONSTRUCTION OF WEARING COURSE

Mixing.—When thoroughly heated to the temperature directed, the asphalt cement or refined tar and the broken stone for the bituminous concrete shall be mixed so that the resulting mixture shall contain between five (5) and eight (8) per cent. by weight of bitumen, as directed, depending primarily upon the kind of bituminous cement and mineral aggregate which are used. A mixer shall be used, having revolving blades, and so designed and operated as to produce and discharge a thoroughly coated and uniform mixture of non-segregated broken stone and bituminous cement. When discharged, mixtures of asphalt cement and broken stone shall have a temperature not more than 149°C. (300°F.) and not less than 93°C. (200°F.) as directed. When discharged, mixtures of refined tar and broken stone shall have a temperature not more than 121°C. (250°F.) and not less than 66°C. (150°F.).

Surface of Foundation.—Before laying the bituminous concrete, the surface of the cement-concrete foundation shall be dry and thoroughly cleaned. If any defective areas exist in the cement-concrete foundation, they shall be repaired as directed at least ten (10) days in advance of laying the bituminous concrete. In cases where cement-concrete edging is not used, compacted shoulders, forming a continuation of the surface of the foundation, shall be constructed. A two (2)-inch plank shall be laid on each shoulder at the edge of the foundation. These planks shall remain in place until after the seal coat has been finished.

Prevention of Dust.—The shoulders of the road and adjacent grounds shall be sufficiently sprinkled during the placing of bituminous material wherever there would otherwise be danger of clouds of dust blowing over the pavement.

Laying Bituminous Concrete.—The bituminous concrete, heated and prepared as specified, shall be delivered direct from the mixer to the point of deposition on the foundation in trucks or wagons, provided with canvas covers for retaining the heat. As delivered the bituminous concrete shall have a temperature of at least 66°C. (150°F.). Material having a lower temperature than this shall not be laid upon the foundation. The hot bituminous concrete shall be dumped upon platforms, constructed as directed, and shoveled with hot shovels into position on the foundation. The bituminous concrete shall be immediately uniformly spread over the foundation course by men experienced in such work and thoroughly compacted by rolling. When compacted the wearing course shall have a thickness at no place of less than two (2) inches and shall be free from surface depressions and irregularities. The paving shall be done as continuously as practicable, to reduce to a minimum the number of joints between hot and cold materials. Such joints shall be constructed as directed.

Rolling.—The bituminous concrete wearing course, laid as specified above, shall be rolled at once, while the mixture is warm and pliable, beginning at the edges and working toward the center. Means for preventing the bituminous material from adhering to the roller without injury to the bituminous concrete, shall be provided. Rolling shall continue without interruption until all roller marks disappear and the surface shows no further compressibility. Places which the roller cannot effectively reach shall be compacted with hot iron tampers.

Roller.—Rollers used on the bituminous concrete shall be well balanced, self-propelled, tandem rollers, weighing between ten (10) and twelve (12) tons each. Each shall have a compression under the rear roller of between two hundred and fifty (250) and three hundred and fifty (350) pounds per linear inch of roll, and shall be provided with an ash pan, which shall prevent ashes from dropping onto the bituminous concrete or seal coat.

Testing Surface.—Before placing the seal coat, the surface of the bituminous concrete shall be tested with a four (4)-foot straight edge laid longitudinally upon any portion of the surface, and any depression or other irregularity exceeding three-eighths (⅜) inch shall be satisfactorily eliminated.

Seal Coat.—As soon as possible after the compaction of the bituminous concrete, when the surface is clean and dry, a seal coat of the hot asphalt cement shall be evenly distributed over the bituminous concrete and uniformly spread by means of squeegees. The asphalt cement shall be applied at a temperature not less than 135°C. (275°F.), nor more than 177°C. (350°F.), at a rate of one-half (½) to one (1) gallon per square yard, as directed. A thin, uniform layer of dry, clean broken stone chips shall be immediately uniformly spread in two applications over the asphalt cement by machines or skilled workmen. Each application of broken stone chips shall be rolled twice by a self-propelled, tandem roller. The spreading of the broken stone chips shall not lag more than twenty (20) feet behind the placing of the asphalt cement coating. Broken stone chips shall not be placed on the wearing course before the asphalt cement of the seal coat is applied. The surface of the bituminous concrete shall be kept scrupulously clean until the seal coat is applied, and the contractor shall not permit any hauling over the wearing course before the completion of the seal coat.

Seasonal and Weather Limitations.—No bituminous concrete shall be mixed or placed when the air temperature in the shade is below 10°C. (50°F.).

PAYMENT

Measurement and Payment.—The quantity of bituminous concrete wearing course to be paid for under this item shall be the number of square yards, measured horizontally, satisfactorily completed in accordance with the specifications. The price stipulated in this item shall include the furnishing, crushing and screening of the broken stone, including the broken stone chips for the seal coat, and heating, mixing, placing and rolling of the broken stone and the asphalt cement or refined tar, and all work and expenses incidental to the completion of the bituminous concrete and the seal coat, except the furnishing of the bituminous cement, which will be included for payment under the item "Asphalt Cements and Refined Tars for Wearing Course of Bituminous Concrete Pavement" or the items "Asphalt Cements and Refined Tars for Wearing Course of Bituminous Concrete Pavement" and "Asphalt Cements for Seal Coat for Bituminous Concrete Pavement." Measurement under this item shall not include any pavement repaired or relaid, except as provided for in the following paragraph.

Removing and Replacing Wearing Course.—If the contractor removes, as directed, portions of the wearing course, and the work thus exposed for examination is found satisfactory, or if for any reason he shall be ordered to remove wearing course built in full accordance with his contract, he shall be paid for such excavation one-fourth the price per square yard stipulated in this item. If the wearing course after examination is found to be of acceptable quality, the original wearing course will be paid for as well as that used to refill the excavation. In connection with the removing and replacing of the wearing course in accordance with this paragraph, no quantity shall be measured as less than one square yard.

SPECIFICATIONS FOR ASPHALT CEMENTS AND REFINED TARS FOR WEARING COURSE OF BITUMINOUS CONCRETE PAVEMENT AND FOR ASPHALT CEMENTS FOR THE SEAL COAT IF AN ASPHALT CEMENT IS USED IN THE BITUMINOUS WEARING COURSE

Asphalt Cement "A" Optional With Asphalt Cements "B," "C," "D," "E" and Refined Tars "F" and "G".

(1) The asphalt cement shall be homogeneous, free from water and shall not foam when heated to 177°C. (350°F.).

(2) It shall show a flash point of not less than 205°C. (400°F.).

(3) Its specific gravity at a temperature of 25°C. (77°F.) shall be not less than 0.970 nor more than 1.000.

(4) When tested with a standard No. 2 needle by means of a Dow penetrometer (or other penetrometer giving the same results as the Dow machine), it shall show penetrations within the following limits for the conditions stated, the penetrations being expressed in hundredths of a centimeter: 100-gram load, 5 seconds, at 25°C. (77°F.), from 75 to 90; 200-gram load, 1 minute, at 4°C. (39°F.), not less than 35; 50-gram load, 5 seconds, at 46°C. (115°F.), not more than 250.

(5) Its melting-point as determined by the cube method shall be not less than 55°C. (131°F.).

(6) When 50 grams of the material is maintained at a uniform temperature of 163°C. (325°F.) for 5 hours in an open cylindrical tin dish, 5½ centimeters (about 2¼ in.) in diameter, with vertical sides measuring approximately 3½ centimeters (about 1½ in.) in depth, the loss in weight shall not exceed 1.0 per cent. of the original weight of the sample.

The penetration of the residue, when tested as described in clause (4) with a standard No. 2 needle under a load of 100 grams, for 5 seconds at 25°C. (77°F.) shall be not less than one-half the penetration of the original material tested under the same conditions.

(7) Its bitumen as determined by its solubility in chemically pure carbon disulphide at room temperature shall not be less than 99.5 per cent.

(8) It shall be soluble in chemically pure carbon tetrachloride at room temperature to the extent of not less than 99.5 per cent. of its bitumen as determined by clause (7).

(9) It shall be soluble in 86° to 88° Bé. paraffin naphtha, at least 85 per cent. distilling between 40° and 55°C. (104° and 131°F.), to the extent of not less than 70.0 per cent. nor more than 80.0 per cent. of its bitumen as determined by clause (7).

(10) It shall yield not less than 8.0 per cent. nor more than 12.0 per cent. of fixed carbon.

Asphalt Cement "B" Optional With Asphalt Cements "A," "C," "D," "E," and Refined Tars "F" and "G"

(1) The asphalt cement shall be homogeneous, free from water and shall not foam when heated to 177°C. (350°F.).

(2) It shall show a flash point of not less than 205°C. (400°F.).

(3) Its specific gravity at a temperature of 25°C. (77°F.) shall be not less than 1.000 nor more than 1.030.

(4) When tested with a standard No. 2 needle by means of a Dow penetrometer (or other penetrometer giving the same results as the Dow machine), it shall show penetrations within the following limits for the conditions stated, the penetrations being expressed in hundredths of a centimeter: 100-gram load, 5 seconds, at 25°C. (77°F.), from 90 to 100; 200-gram load, 1 minute at 4°C. (39°F.), not less than 30.

(5) Its melting-point as determined by the cube method shall be not less than 50°C. (122°F.).

(6) When 50 grams of the material is maintained at a uniform temperature of 163°C. (325°F.) for 5 hours in an open cylindrical tin dish, 5½ centimeters (about 2¼ in.) in diameter, with vertical sides measuring approximately 3½ centimeters (about 1½ in.) in depth, the loss in weight shall not exceed 1.0 per cent. of the original weight of the sample.

The penetration of the residue, when tested as described in clause (4) with a standard No. 2 needle under a load of 100 grams, for 5 seconds at 25°C. (77°F.) shall be not less than one-half the penetration of the original material tested under the same conditions.

(7) Its bitumen as determined by its solubility in chemically pure carbon disulphide at room temperature shall not be less than 99.5 per cent.

(8) It shall be soluble in chemically pure carbon tetrachloride at room temperature to the extent of not less than 99.5 per cent. of its bitumen as determined by clause (7).

(9) It shall be soluble in 86° to 88° Bé. paraffin naphtha, at least 85 per cent. distilling between 40° and 55°C. (104° and 131°F.), to the extent of not less than 72.0 per cent. nor more than 78.0 per cent. of its bitumen as determined by clause (7).

(10) It shall yield not less than 11.0 per cent. nor more than 15.0 per cent. of fixed carbon.

Asphalt Cement "C" Optional With Asphalt Cements "A," "B," "D," "E" and Refined Tars "F" and "G"

(1) The asphalt cement shall be homogeneous, free from water and shall not foam when heated to 177°C. (350°F.).

(2) It shall show a flash point of not less than 205°C. (400°F.).

(3) Its specific gravity at a temperature of 25°C. (77°F.) shall be not less than 1.030 nor more than 1.040.

(4) When tested with a standard No. 2 needle by means of a Dow penetrometer (or other penetrometer giving the same results as the Dow machine), it shall show penetrations within the following limits for the conditions stated, the penetrations being expressed in hundredths of a centimeter: 100-gram load, 5 seconds, at 25°C. (77°F.), from 70 to 90; 200-gram load, 1 minute, at 4°C. (39°F.), not less than 10.

(5) Its melting point as determined by the cube method shall be not less than 45°C. (113°F.).

(6) When 50 grams of the material is maintained at a uniform temperature of 163°C. (625°F.) for 5 hours in an open cylindrical tin dish, 5½ centimeters (about 2¼ in.) in diameter, with vertical sides measuring approximately 3½ centimeters (about 1½ in.) in depth, the loss in weight shall not exceed 2.0 per cent. of the original weight of the sample.

The penetration of the residue, when tested as described in clause (4) with a standard No. 2 needle under a load of 100 grams, for 5 seconds at 25°C. (77°F.) shall be not less than one-half the penetration of the original material tested under the same conditions.

(7) Its bitumen as determined by its solubility in chemically pure carbon disulphide at room temperature shall not be less than 99.5 per cent.

(8) It shall be soluble in chemically pure carbon tetrachloride at room temperature to the extent of not less than 99.5 per cent. of its bitumen as determined by clause (7).

(9) It shall be soluble in 86° to 88° Bé. paraffin naphtha, at least 85 per cent. distilling between 40° and 55°C (104° and 131°F.), to the extent of not less than 80.0 per cent. nor more than 88.0 per cent. of its bitumen as determined by clause (7).

(10) It shall yield not less than 10.0 per cent. nor more than 14.0 per cent. of fixed carbon.

Asphalt Cement "D" Optional With Asphalt Cements "A," "B," "C," "E," and Refined Tars "F" and "G"

(1) The asphalt cement shall be homogeneous, free from water and shall not foam when heated to 177°C. (350°F.).

(2) It shall show a flash point of not less than 205°C. (400°F.).

(3) Its specific gravity at a temperature of 25°C. (77°F.) shall be not less than 1.025 nor more than 1.050.

(4) When tested with a standard No. 2 needle by means of a Dow penetrometer (or other penetrometer giving the same results as the Dow machine), it shall show penetrations within the following limits for the conditions stated, the penetrations being expressed in hundredths of a centimeter: 100-gram load, 5 seconds, at 25°C. (77°F.), from 85 to 95; 200-gram load, 1 minute, at 4°C. (39°F.), not less than 20.

(5) Its melting-point as determined by the cube method shall be not less than 50°C. (122°F.).

(6) When 50 grams of the material is maintained at a uniform temperature of 163°C. (325°F.) for 5 hours in an open cylindrical tin dish, 5½ centimeters (about 2¼ in.) in diameter, with vertical sides measuring approximately 3½ centimeters (about 1½ in.) in depth, the loss in weight shall not exceed 2.0 per cent. of the original weight of the sample.

The penetration of the residue, when tested as described in clause (4) with a standard No. 2 needle under a load of 100 grams, for 5 seconds at 25°C. (77°F.) shall be not less than one-half the penetration of the original material tested under the same conditions.

(7) Its bitumen as determined by its solubility in chemically pure carbon disulphide at room temperature shall not be less than 99.5 per cent.

(8) It shall be soluble in chemically pure carbon tetrachloride at room temperature to the extent of not less than 99.5 per cent. of its bitumen as determined by clause (7).

(9) It shall be soluble in 86° to 88° Bé. paraffin naphtha, at least 85 per cent. distilling between 40° and 55°C. (104° and 131°F.), to the extent of not less than 67.0 per cent. nor more than 77.0 per cent. of its bitumen as determined by clause (7).

(10) It shall yield not less than 12.0 per cent. nor more than 18.0 per cent. of fixed carbon.

Asphalt Cement "E" Optional With Asphalt Cements "A," "B," "C," "D," and Refined Tars "F" and "G"

(1) The asphalt cement shall be homogeneous, free from water and shall not foam when heated to 177°C. (350°F.).

(2) It shall show a flash point of not less than 165°C. (329°F.).

(3) Its specific gravity at a temperature of 25°C. (77°F.) shall be not less than 1.040 nor more than 1.060.

(4) When tested with a standard No. 2 needle by means of a Dow penetrometer (or other penetrometer giving the same results as the Dow machine) it shall show penetrations within the following limits for the conditions stated, the penetrations being expressed in hundredths of a centimeter: 100-gram load, 5 seconds, at 25°C. (77°F.), from 104 to 160; 200-gram load, 1 minute, at 4°C. (39°F.), not less than 40.

(5) When tested by means of the New York Testing Laboratory Float Apparatus, the float shall not sink in water maintained at 66°C. (150°F.) in less than 120 seconds nor more than 180 seconds.

(6) When 50 grams of the material is maintained at uniform temperature of 163°C. (325°F.) for 5 hours in an open cylindrical tin dish, 5½ centimeters (about 2¼ in.) in diameter, with vertical sides measuring approximately 3½ centimeters (about 1½ in.) in depth, the loss in weight shall not exceed 3.0 per cent. of the original weight of the sample.

The penetration of the residue, when tested as described in clause (4) with a standard No. 2 needle under a load of 100 grams, for 5 seconds at 25°C. (77°F.) shall be not less than one-half the penetration of the original material tested under the same conditions.

(7) Its bitumen as determined by its solubility in chemically pure carbon disulphide at room temperature shall be not less than 93.0 per cent. nor more than 98.0 per cent.

(8) It shall be soluble in chemically pure carbon tetrachloride at room temperature to the extent of not less than 98.5 per cent. of its bitumen as determined by clause (7).

(9) It shall be soluble in 86° to 88° Bé. paraffin naphtha, at least 85 per cent. distilling between 40° and 55°C. (104° and 131°F.), to the extent of not less than 75.0 per cent. nor more than 85.0 per cent. of its bitumen as determined by clause (7).

(10) It shall yield not less than 11.0 per cent. nor more than 15.0 per cent. of fixed carbon.

(11) Upon ignition it shall yield not less than 1.0 per cent. nor more than 3.0 per cent. of ash.

Refined Tar "F" Optional With Asphalt Cements "A," "B," "C," "D," "E," and Refined Tar "G"

(1) The refined tar shall be homogeneous, free from water and shall not foam when heated to 150°C. (302°F.).

(2) Its specific gravity at a temperature of 25°C. (77°F.) shall be not less than 1.160 nor more than 1.200.

(3) When tested by means of the New York Testing Laboratory Float Apparatus, the float shall not sink in water maintained at 50°C. (122°F.) in less than 140 seconds nor more than 170 seconds.

(4) Its bitumen as determined by its solubility in chemically pure carbon disulphide at room temperature shall be not less than 95.0 per cent. and it shall show not more than 0.2 per cent. ash upon ignition of the material insoluble in carbon disulphide.

(5) When distilled according to the tentative methods recommended by Committee D-4 of the American Society for Testing Materials in 1911, it shall yield no distillate at a temperature lower than 170°C. (338°F.); not more than 7.0 per cent. by weight shall distil below 270°C. (518°F.), and not more than 20.0 per cent. by weight shall distil below 300°C. (572°F.).

(6) The total distillate from the test made in accordance with clause (5) shall have a specific gravity at a temperature of 25°C. (77°F.) of not less than 1.000 nor more than 1.020.

(7) The melting-point, as determined in water by the cube method, of the pitch residue

remaining after distillation to 300°C. (572°F.), in accordance with the test described in clause (5), shall be not more than 75°C. (167°F.).

Refined Tar "G" Optional With Asphalt Cements "A," "B," "C," "D," "E," and Refined Tar "F"

(1) The refined tar shall be homogeneous, free from water and shall not foam when heated to 150°C. (302°F.).

(2) Its specific gravity at a temperature of 25°C. (77°F.) shall be not less than 1.200 nor more than 1.300.

(3) When tested by means of the New York Testing Laboratory Float Apparatus, the float shall not sink in water maintained at 50°C. (122°F.) in less than 140 seconds nor more than 170 seconds.

(4) Its bitumen as determined by its solubility in chemically pure carbon disulphide at room temperature shall be not less than 75.0 per cent. nor more than 90.0 per cent., and it shall not show more than 0.2 per cent. ash upon ignition of the material soluble in carbon disulphide.

(5) When distilled according to the tentative method recommended by Committee D-4 of the American Society for Testing Materials in 1911, it shall yield no distillate at a temperature lower than 170°C. (338°F.); not more than 10.0 per cent. by weight shall distil below 270°C. (518°F.), and not more than 20.0 per cent. by weight shall distil below 300°C. (572°F.)

(6) The total distillate from the test made in accordance with clause (5) shall have a specific gravity at a temperature of 25°C. (77°F.) of not less than 1.030.

(7) The melting point, as determined in water by the cube method, of the pitch residue remaining after distillation to 300°C. (572°F.), in accordance with the test described in clause (5), shall be not more than 75°C. (167°F.).

SPECIFICATIONS FOR ASPHALT CEMENTS FOR SEAL COAT FOR BITUMINOUS CONCRETE PAVEMENT WHEN REFINED TAR IS USED IN THE BITUMINOUS CONCRETE WEARING COURSE

Asphalt Cement "H" Optional With Asphalt Cements "K" and "L"

(1) The asphalt cement shall be homogeneous, free from water and shall not foam when heated to 177°C. (350°F.).

(2) It shall show a flash point of not less than 205°C. (400°F.)

(3) Its specific gravity at a temperature of 25°C. (77°F.) shall be not less than 0.970 nor more than 1.000.

(4) When tested with a standard No. 2 needle by means of a Dow penetrometer (or other penetrometer giving the same results as the Dow machine), it shall show penetrations within the following limits for the conditions stated, the penetrations being expressed in hundredths of a centimeter: 100-gram load, 5 seconds, at 25°C. (77°F.), from 60 to 75; 200-gram load, 1 minute, at 4°C. (39°F.), not less than 50; 50-gram load, 5 seconds, at 46°C. (115°F.), not more than 150.

(5) Its melting-point as determined by the cube method shall be not less than 80°C. (176°F.).

(6) When 50 grams of the material is maintained at a uniform temperature of 163°C. (325°F.) for 5 hours in an open cylindrical tin dish, 5½ centimeters (about 2¼ in.) in diameter, with vertical sides measuring approximately 3½ centimeters (about 1½ in.) in depth, the loss in weight shall not exceed 1.0 per cent. of the original weight of the sample.

The penetration of the residue, when tested as described in clause (4) with a standard No. 2 needle under a load of 100 grams, for 5 seconds at 25°C. (77°F.) shall be not less than one-half the penetration of the original material tested under the same conditions.

(7) Its bitumen as determined by its solubility in chemically pure carbon disulphide at room temperature shall not be less than 99.5 per cent.

(8) It shall be soluble in chemically pure carbon tetrachloride at room temperature to the extent of not less than 99.5 per cent. of its bitumen as determined by clause (7).

(9) It shall be soluble in 86° to 88° Bé. paraffin naphtha, at least 85 per cent. distilling between 40° and 55°C. (104° and 131°F.), to the extent of not less than 70.0 per cent. nor more than 80.0 per cent. of its bitumen as determined by clause (7).

(10) It shall yield not less than 8.0 per cent. nor more than 12.0 per cent. of fixed carbon.

Asphalt Cement "K" Optional With Asphalt Cements "H" and "L"

(1) The asphalt cement shall be homogeneous, free from water and shall not foam when heated to 177°C. (350°F.).

(2) It shall show a flash point of not less than 205°C. (400°F.).

(3) Its specific gravity at a temperature of 25°C. (77°F.) shall be not less than 1.030 nor more than 1.045.

(4) When tested with a standard No. 2 needle by means of a Dow penetrometer (or other penetrometer giving the same results as the Dow machine), it shall show penetrations within the following limits for the conditions stated, the penetrations being expressed in hundredths of a centimeter: 100-gram load, 5 seconds, at 25°C. (77°F.), from 60 to 70; 200-gram load, 1 minute, at 4°C. (39°F.), not less than 18; 50-gram load, 5 seconds, at 46°C. (115°F.), not more than 270.

(5) Its melting-point as determined by the cube method shall be not less than 60°C. (140°F.).

(6) When 50 grams of the material is maintained at a uniform temperature of 163°C. (325°F.) for 5 hours in an open cylindrical tin dish, 5½ centimeters (about 2¼ in.) in diameter, with vertical sides measuring approximately 3½ centimeters (about 1½ in.) in depth, the loss in weight shall not exceed 1.0 per cent. of the original weight of the sample.

The penetration of the residue, when tested as described in clause (4) with a standard No. 2 needle under a load of 100 grams, for 5 seconds at 25°C. (77°F.) shall be not less than one-half the penetration of the original material tested under the same conditions.

(7) Its bitumen as determined by its solubility in chemically pure carbon disulphide at room temperature shall not be less than 99.5 per cent.

(8) It shall be soluble in chemically pure carbon tetrachloride at room temperature to the extent of not less than 99.5 per cent. of its bitumen as determined by clause (7).

(9) It shall be soluble in 86° to 88° Bé. paraffin naphtha, at least 85 per cent. distilling between 40° and 55°C. (104° and 131°F.), to the extent of not less than 70.0 per cent. nor more than 80.0 per cent. of the bitumen as determined by clause (7).

(10) It shall yield not less than 12.0 per cent. nor more than 16.0 per cent. of fixed carbon.

Asphalt Cement "L" Optional With Asphalt Cements "H" and "K"

(1) The asphalt cement shall be homogeneous, free from water and shall not foam when heated to 177°C. (350°F.).

(2) It shall show a flash point of not less than 205°C. (400°F.).

(3) Its specific gravity at a temperature of 25°C. (77°F.) shall be not less than 1.025 nor more than 1.055.

(4) When tested with a standard No. 2 needle by means of a Dow penetrometer (or other penetrometer giving the same results as the Dow machine), it shall show penetrations within the following limits for the conditions stated, the penetrations being expressed in hundredths of a centimeter: 100-gram load, 5 seconds, at 25°C. (77°F.), from 60 to 70; 200-gram load, 1 minute, at 4°C. (39°F.), not less than 16.

(5) Its melting-point as determined by the cube method shall be not less than 55°C. (131°F.).

(6) When 50 grams of the material is maintained at a uniform temperature of 163°C. (325°F.) for 5 hours in an open cylindrical tin dish, 5½ centimeters (about 2¼ in.) in diameter, with vertical sides measuring approximately 3½ centimeters (about 1½ in.) in depth, the loss in weight shall not exceed 1.0 per cent. of the original weight of the sample.

The penetration of the residue, when tested as described in clause (4) with a standard No. 2 needle under a load of 100 grams, for 5 seconds at 25°C. (77°F.) shall be not less than one-half the penetration of the original material tested under the same conditions.

(7) Its bitumen as determined by its solubility in chemically pure carbon disulphide at room temperature shall not be less than 99.5 per cent.

(8) It shall be soluble in chemically pure carbon tetrachloride at room temperature to the extent of not less than 99.5 per cent. of its bitumen as determined by clause (7).

(9) It shall be soluble in 86° to 88° Bé. paraffin naphtha, at least 85 per cent. distilling between 40° and 55°C. (104° and 131°F.), to the extent of not less than 67.0 per cent. nor more than 77.0 per cent. of its bitumen as determined by clause (7).

(10) It shall yield not less than 13.0 per cent. nor more than 18.0 per cent. of fixed carbon.

Delivery.—The asphalt cement or refined tar shall be delivered in suitable containers,

far enough in advance of its use in the work to permit the necessary tests to be made. Each container shall be plainly labeled with the trade name of the asphalt cement or refined tar, name of manufacturer, gross weight and net weight. Each shipment and each carload shall be kept separate.

Bills of Lading.—The contractor shall furnish the engineer on or before the arrival of each shipment at or near the site of the work, bills of lading, or correct copies thereof, which shall state the trade name of the asphalt cement or refined tar, and the name and address of the company manufacturing and supplying it.

Samples. –Samples will be taken by the engineer from each carload of asphalt cement or refined tar when delivered at the work, unless satisfactory arrangements can be made for sampling before shipment. Such samples shall be analyzed by the engineer to assure the delivery of an asphalt cement or refined tar of the specified quality and to determine, for purpose of payment, the quantity of bitumen.

Work Included.—Under this item the contractor shall furnish and deliver on the work at such points as directed an asphalt cement or an asphalt cement and a refined tar, which conform with the specifications heretofore mentioned. All asphalt cement or refined tar for any pavement of a given contract shall be furnished by one manufacturer and no change in type or grade of bituminous cement used in the bituminous concrete or the seal coat shall be allowed in any pavement of a given contract without written permission from the engineer.

Measurement and Payment.—The quantity of bitumen in the asphalt cement or refined tar, to be paid for under this item, shall be the number of tons, determined in accordance with the paragraph headed "Samples," contained in the asphalt cement or refined tar placed in the pavement in accordance with the specifications and requirements, or used as directed for other purposes. The percentage of bitumen determined by an average of the analyses of the acceptable samples taken by the engineer during a given month, shall be used as the basis for payment for the asphalt cement or refined tar used during that month. Asphalt cement or refined tar that is wasted shall not be included in the measurement under this item. The price stipulated in this item shall include the cost of furnishing, hauling and delivering the asphalt cement or refined tar at points on the work where it is to be used, and all expenses incidental thereto.

Final Report of the Special Committee of the American Society of Civil Engineers on Materials for Road Construction and on Standards for Their Test and Use. (From Trans. Am. Soc. Civ. Eng., 82 (1918), 1384.)

FINAL REPORT OF THE SPECIAL COMMITTEE ON MATERIALS FOR ROAD CONSTRUCTION AND ON STANDARDS FOR THEIR TEST AND USE[1]

With Discussion by Messrs. E. Dow Gilman and J. O. Preston

To the American Society of Civil Engineers,
Gentlemen:

Your Special Committee on "Materials for Road Construction and on Standards for Their Test and Use" herewith respectfully submits a Final Report covering its work from the date of its appointment by the Board of Direction.

Through the courtesy of the Board, successful meetings have been held in the Society House, for one or two days subsequent to each Annual Meeting of the Society, for the discussion of matters of interest connected with the work of the Committee. These meetings have been well attended, usually by more than 200 at a session, by highway authorities outside of the Society as well as by members of the Society itself. It is felt that valuable, as well as authoritative, information and criticism has thus been secured to the Profession as well as to the Committee.

Your Committee has rendered progress reports annually since 1911, and in the accompanying report has given careful consideration to its earlier conclusions and the criticism and discussions submitted in connection therewith.

The Committee has been made aware of a widening of the interest in highway work

[1] Presented to the Annual Meeting, January 16, 1918.

among members of the Society and others not experienced or even thoroughly trained in-highway engineering. In the accompanying report it has been deemed wise, for the benefit of this new and wider interest, as well as for the purpose of making this Final Report comprehensive, to include conclusions or statements that may seem primary or trite to highway experts. Further, in order to render many of the earlier and present conclusions of the Committee more intelligible to the wider interest mentioned, as well as to demonstrate their practical application in many cases, and to suggest the variables still remaining to be determined in specific instances, it has been thought wise to submit in this report principles underlying the drafts of specifications.

Your Committee believes that competent highway engineers may meet successfully the demands of any particular case by following these principles and eliminating the variables, necessarily left therein in order to express conclusions of general application, after proper consideration of the local factors affecting them.

However, it does not believe that these conclusions as to specifications will offset a serious lack of knowledge or experience in highway engineering or furnish a discriminating ability otherwise lacking. It also wishes to warn against any attempt to reduce the principles for specifications to the establishment of "the one best pavement" for any conditions.

One of the main activities of the Committee since its foundation has been toward securing accuracy and uniformity in the recording of data connected with highway work. To this end the Committee has suggested tests and analyses of materials in detail, records of traffic, costs, construction and maintenance details, and definitions of terms used with peculiar meanings in highway work. When it began its work in 1909, the status of affairs in these respects was found to be deplorable. Since then the work of this Committee and of committees on similar lines from other societies, as well as the work of some individuals, has materially improved the situation. Your Committee has made some suggestions which have been quite widely adopted. On the other hand, it has not hesitated to endorse the conclusions of others where such action seemed proper, hoping thereby to aid materially in the accomplishment of that desirable uniformity mentioned above. It hopes that its conclusions may be generally approved, even at the sacrifice of some minor differences of opinion, in order that uniformity in these matters, with resulting benefit, may be secured at the earliest possible date. The Forms, Analyses, and Test Details will be found in the Appendices hereto attached.

In its previous reports, your Committee has suggested a number of matters worthy of investigation. Sufficient time has not yet been available to the Committee for reaching satisfactory conclusions on all these points, and it has seemed to be a grave question whether or not it would be able, with the facilities at its disposal, to reach such conclusions within a reasonable period. Your Committee, therefore, invites the attention of all those interested to the following list of investigations it thinks worthy of prosecution, and expresses the hope that some authoritative data, which will throw light on these points, may in some way be secured at an early date:

The effect of various kinds of earth or soil on bituminous carpets under known conditions of traffic;

The efficiency of the use of asphaltic oils for surface treatments on gravel and broken-stone roadways;

The standardization of, and determination and expression of the consistency of, bituminous materials which preferably may permit of its use on all the wide range of such materials, from liquid to solid, and of the inter-relation existing in certain, if not all, cases;

The maximum and minimum quantities of free carbon that can be successfully allowed in tars under known conditions of other constituents of the tar, of climate, of traffic, and of methods of use;

Methods of determining the adhesive strength of bituminous cements;

The advisability of incorporating in bituminous pavements, built by the penetration method, bituminous material in excess of the minimum quantity necessary for the production of satisfactory results;

The determination of the amount of internal wear of the materials comprising a macadam roadway under known conditions of traffic, and the effects on the internal wear of increasing or diminishing the size of the stone in the courses;

The determination of the causes of cracks in concrete and brick roadways, and the best methods to be used to reduce such cracks to a minimum;

Rolling or other methods calculated to increase the surface density of cement-concrete pavements;

The relative merits of one-course and two-course plain cement-concrete pavements, and plain and reinforced cement-concrete pavements;
The relative efficiency of bituminous and cement fillers for block pavements;
The determination of the quality—which might be called its hardness or resiliency—of wood used for paving purposes;
The continuance of traffic census, and study of the results obtained, to determine the effect of motor traffic on various roadway surfaces, and especially with reference to the speed of the vehicle, and the establishment and expression of any relation between the traffic and the life or cost of any pavement;
The further study of suitable joints for block pavements on steep grades;
Proper methods of sampling highway materials.

Many points are as yet undetermined, and seem improbable of early settlement. Conclusions, in many instances, will be hastened by a prompt agreement among investigators that uniformity is fully as necessary as accuracy in their work, and that the use of methods of analysis conforming to those carefully specified in this report will produce better and quicker results than a wide use of various methods, more or less accurate in some details, but not consonant with those of the Committee. The conclusions of your Committee have been reached and expressed after careful consideration of the work of committees of other societies and with intimate knowledge of the details of that work through the membership on these other committees of members of this Committee.

Your Committee is impressed by the importance of the factor of costs, both as to construction and maintenance of highways, and of the need for comparable records of such data. The present situation in this respect is unfortunate, there being found available but few records of costs uniformly or logically compiled. Hence comparisons are difficult, if not impossible. Your Committee, therefore, invites especial attention to its conclusions and forms in the matters of costs. The same may be said concerning traffic records. The propriety of a material or a form of highway construction is often determined by its cost under known traffic conditions. To reduce both cost and traffic data in different cases for purposes of comparison requires that they be expressed in some uniform, definite, and intelligible manner.

Your Committee submits the specific conclusions it has reached, as follows:

GENERAL CONCLUSIONS

Selection of Roadway Surface.—The Committee believes that, with the development of highway work, it should be constantly more apparent that one of the greatest problems to be solved by highway engineers is the proper selection of the particular material and form of construction to be used which will most efficiently meet the conditions of any particular case, and that progress will be hastened by complete recognition of this fact. It recommends that the selection of the kind of crust or pavement be based on the following factors, the special value of which may be estimated in each case under the local conditions of traffic, surroundings, climatic conditions, and physical and financial resources, both as to construction and maintenance, with proper regard for probable or possible changes in these circumstances: First cost, maintenance cost, annual cost, ease of maintenance, durability, cleanliness, tractive resistance, slipperiness, favorableness to travel, sanitariness, noiselessness, and appearance.

Traffic.—Your Committee desires to emphasize the fact that experience has demonstrated the value of a traffic census taken both preliminary and subsequent to the construction of a highway. The traffic census should be considered one of the most important variable factors in the solution of the problem of the selection of that type of construction best suited to local conditions, considered from the standpoints of both economy and efficiency. In connection with the census returns on any highway, should be considered the traffic on cross and parallel highways and the effect of improvement of these highways on the traffic of the highway under construction. The bald return of a traffic census, however, should not be the sole basis of the selection of the type of construction, but should be considered a guide in determining the value of the type to be adopted. In considering the effect of traffic and its relation to the design and cost of maintenance, it is necessary to take into account the speed as well as the weight of the vehicles.

Costs.—The Committee recommends that highway departments, in making their reports, adopt a tabular form similar to the one submitted on page 464.

Grades.—A choice of the material, or methods of using a particular material, may be

affected by the grades as fixed. Certain materials or results of using materials for highway surfacings will be unsatisfactory outside of certain limits of grades. Conservative practice fixes the maximum limits for satisfactory results with grades as follows:

Kind of roadway		Maximum grade
Asphalt block		8.0 per cent.
Bituminous surfaces		6.0 per cent.
Bituminous concrete		8.0 per cent.
Bituminous macadam		8.0 per cent.
Brick	cement filler	6.0 per cent.
	bituminous filler	12.0 per cent.
	"Hillside" block	15.0 per cent.
Broken stone		12.0 per cent.
Cement-concrete		8.0 per cent.
Gravel		12.0 per cent.
Sheet-asphalt		5.0 per cent.
Stone block	cement filler	9.0 per cent.
	bituminous filler	15.0 per cent.
Wood block		4.0 per cent.

The minimum grades allowable will depend on local conditions as to climate, type of construction, character and amount of traffic, conditions of underlying and adjacent soil, and such other circumstances as affect drainage. Except for roadways on fills, where the outside edges of the surfaces of the shoulders are at least 2 ft. above the level of the adjacent ground or water level, or except in cases where the roadway is laid over sand of such a character that it never becomes water-logged, a longitudinal grade for the roadway of less than one-half of 1 per cent. should not be used for roads.

On streets where the smoothness and evenness of the roadway surface may be confidently expected to be the greatest, and where conformity to the proposed elevations of surface is more carefully sought and more accurately possible, a minimum grade of as low as one-quarter of 1 per cent. has sometimes given satisfactory results in an emergency; but a minimum of one-half of 1 per cent. would be better as an established standard.

Widths.—The width of the roadway to be built will be determined largely by local circumstances, but, in view of the recent, constant, and rapid increase of traffic on highways, both in number of vehicles and in size of loads, it will be in the interest of economy for designs of highways to be made with proper consideration of further increase.

Where motor traffic forms a considerable proportion of the total traffic likely to use a highway, the unit width of traffic lines to be considered is 9 or 10 ft., instead of 7 or 8 ft., as heretofore, because of greater clearance required for the safe passing of the units of such traffic.

Where bituminous pavements are laid, the edges need protection, and a sudden transition from the pavement to any soft shoulder material should be avoided by means of extra width, or of cement-concrete or other edges, or such reinforcement of the shoulder material as may be necessary.

The width of roadways of rigid material, such as cement-concrete or vitrified block, should be at least equal to what would be prescribed under local conditions for a less rigid surfacing. The great difference between the firmness of a rigid roadway surfacing and of material frequently available for the shoulders therero, often makes it necessary for safety and convenience of traffic, as well as for economy of maintenance, that the rigid surfacing shall be built wider than would answer for a more flexible surfacing, such as water-bound macadam, for instance, under the same local conditions. For single-track roadways, the width of the pavement should not be less than 10 ft., and for two lines of traffic, it should not be less than 18 ft., unless exceptionally durable shoulders are provided. In a street or alley, the width will ordinarily be determined by the necessary location of the curb.

Too narrow a width of roadway encourages, if it does not compel, concentration of traffic to such an extent as frequently to make unfair demands on what would otherwise be a suitable and efficient material for the surfacing. This may be especially noticeable at abrupt changes in the lines of the highway, where any tendency toward the improper concentration of traffic into too narrow areas should be avoided, as far as possible, by such adjustment or separation of lines, and adjustment of width, of crown, or of slope, of the roadway surfacing as will keep the strains of the surfacing material within reasonable limits.

Too great a width for the roadway surfacing is as unwise in many ways as too little. Excessive width not only results in unnecessary first cost and interest charges, but also in needlessly increased maintenance and cleaning costs. Further, especially in the cases of those pavements where at least a minimum amount of travel is needed to preserve the surface in good condition, an excess of width may result in the development of areas from which disintegration of the whole pavement may rapidly spread.

Thickness.—In determining the thickness of any road crust or pavement, there must be consideration of the character of the foundation and of the weight of the vehicles to be supported. Although the general practice has been, too often, perhaps, to use mass, for the sake of safety in the preparation of the pavement, it now appears evident that some waste has been incurred in this direction, and that a more scientific determination of the thickness is possible without sacrifice of safety and yet with economy. However, in view of the recent, constant, and rapid increase of the weight, and consequently of the strains, caused by the traffic, it will be in the interests of economy for all designs of highways to be made with proper consideration of further increases.

In considering the character and capabilities of the foundation relative to the forces coming thereon through a pavement, the condition of the foundation under the most adverse conditions likely to exist for withstanding the forces, and the traffic stresses present and probable, and the character of the surfacing itself, should be taken into account. An absorbent sub-grade material likely to become soaked with water so as to weaken its supporting power may require a thicker slab, or even the addition of an artificial foundation, in order to disperse properly the stresses from the surface of the pavement. For instance, if laid on a strong sub-grade and one likely to remain always in good condition, the minimum thickness of 6 in. for a broken stone roadway will be sufficient, and possibly even 4 in. will be enough where the vehicles passing over the road are comparatively light, that is, of 1 or 2 tons on four wheels. With a concrete slab, ordinarily, from 4 to 8 in. in thickness will suffice, though, in order to prevent the possibility of a sudden rupture of the slab on some sub-grades by an exceptional load, a uniform thickness above the minimum may be wise. Again, mass—that is, unusual thickness—may in some cases be desirable with the use of a minimum of cement, not only for reasons of economy, but also for the purpose of avoiding the ill effects of frost and possibly of preparing for future developments that may seem probable. A thickness for a concrete slab in excess of 8 in. should be determined upon only after thoroughly considering the possibilities of meeting the necessities of the case by other means, such as improving the sub-grade.

Although the distribution by the road crust or pavement of the traffic stresses through it to the foundation is not at present within the possibilities of calculation in the case of all types of pavements, progress has been, and is being, made along these lines. Logical and fairly accurate formulas have been developed in the case of broken stone road crusts, and studies productive of some results have been made in the case of some other surfacings. Also, the bearing power of soils of different kinds has been given considerable study. These studies should be encouraged and carried on, so that the thickness of the road crust or pavement for any type may be rationally determined.

Uniformity of thickness for the surfacing is made for the purpose of conducing toward uniformity of wear. Variations in the thickness of such surfacings as sheet-asphalt, for instance, invariably result in non-uniformity of wear, with a resulting increase of expense for satisfactory maintenance; and it is believed that the same cause is responsible for many of the difficulties experienced in the maintenance of block pavements of various types. The development of depressions in the surfaces of these pavements and the deterioration of areas of the pavement seem to be explained by the irregularities in the thickness of the surfacing or of the cushion or foundation under the surfacing over such areas, which irregularities produce unequal settlement or decided differences in rigidity.

The thickness of the pavement or surfacing, of course, will be dependent largely on its type. Approved practice establishes the limits shown in Table 1 for the extremes of thickness for the various layers of the pavement or road crust.

Drainage.—The use of any form of pavement or road crust, whether bituminous or non-bituminous, does not relieve the necessity for proper drainage in every case. It is not only necessary to provide for such under-drainage as will place and keep the sub-grade in a condition satisfactorily free from moisture and in a state of suitable efficiency, but it is also necessary to provide and to preserve economically such provisions for surface drainage as will, with the provisions for under-drainage, insure these results fairly permanently. Storm-water coming to the roadway must be carried quickly and rapidly away from it by auto-

TABLE 1

Kind of roadway	Thickness of artificial foundation[1] (ordinary), in inches	Thickness of sand cushion or binder course, in inches	Thickness of wearing course, in inches
Asphalt block	5 to 8	.....	2 to 3½
Bituminous surfaces	4 to 8	.....	¼ to ½
Bituminous concrete	3 to 8	.....	1½ to 3
Bituminous macadam	3 to 8	.....	2 to 3
Brick	4 to 8	¾ to 1½	3 to 4
Broken stone	3 to 8	.....	2 to 3
Cement-concrete { One course	.....	.....	5 to 8
Cement-concrete { Two course	4 to 8	.....	2
Gravel	4 to 8	.....	2 to 4
Sheet-asphalt	5 to 8	1 to 1½	1½ to 2
Stone block	5 to 12	1 to 2	2½ to 5
Wood block	5 to 8	0 to ½	3½ to 4

[1] Not including extraordinary provisions such as V-drains or "sub-base" courses.

matic arrangements to the natural watercourses where it can be finally disposed of. The arrangements referred to and so made, such as inlets, ditches, gutters, and culverts, should be designed and placed so as give the least possible offense to the users of the roadway and the abuttors, and yet be built so as to preserve their integrity and efficiency with the least need for attention and expense, under even the most persistently adverse natural conditions. A proper longitudinal grade for ditches and gutters is particularly important, in order that the ill effects of standing water may be avoided. A proper cross-section for ditches is also important, in order that the water may not become obstructed by tne sliding in of the sides.

The under-drainage of the roadbed, where a cement-concrete roadway or an artificial foundation of cement-concrete is to be provided, should be at least as good as that which would be required in most cases of other surfaces, because the rigidity of the cement-concrete slab does not permit it to adapt itself—as is the case, for instance, with such a surface as macadam—without injury to changes in the sub-grade resulting from defective drainage.

As related to drainage, the matter of the crown of the roadway is particularly important. The ideal roadway surface would be flat in cross-section were it not for the necessity for the removal of surface water to the channels where it must be most conveniently carried along. Crowning the roadway tends to concentrate the traffic on the ridge, where it is then most confortable for the travelers, and the amount of crown which will result in this concentration on the ridge varies with the type of pavement. Also, the rate of crown necessary for the proper removal of storm-water to the gutters or ditches varies with the type and with the provisions to be made for the cleaning and the upkeep of the roadway surface. In the general practice, the amount of crown for the shoulders of an uncurbed roadway has usually been a cross-slope of 1 in. per ft., the shoulders being of the natural earthy material, and this rate is to be recommended for shoulders, except in special cases.

With pavements inclined to be slippery under certain conditions the crown should be reduced to the lowest possible minimum consistent with surface drainage; and, where the longitudinal grade is sufficient to allow the water to run off freely, the crown should be very flat—not exceeding 3 in. in a roadway width of 30 ft.

For the various roadway surfacings, the practice generally observed and to be recommended is stated in Table 2.

Concave pavements of cement-concrete, vitrified block, or stone block, may frequently be found advantageous for alleys, and, in such cases, the same rates of slopes in cross-section as those given in Table 2 should govern.

Sub-grade.—The use of any form of pavement or road crust does not relieve the necessity for the construction of a well-drained, thoroughly compacted, homogeneous, and stable sub-grade in every case. Indeed, such improvement of the highway generally attracts heavier traffic, and thus increases the stresses in the sub-grade. Even when an artificial

TABLE 2

Kind of roadway	Maximum	Minimum
Asphalt block	¼ in. to the ft.	⅛ in. to the ft.
Bituminous surfaces	½ in. to the ft.	¼ in. to the ft.
Bituminous concrete	½ in. to the ft.	¼ in. to the ft.
Bituminous macadam	½ in. to the ft.	¼ in. to the ft.
Brick pavements	⅜ in. to the ft.	⅛ in. to the ft.
Broken stone	¾ in. to the ft.	½ in. to the ft.
Cement-concrete	⅜ in. to the ft.	¼ in. to the ft.
Gravel	1 in. to the ft.	½ in. to the ft.
Sheet-asphalt	¼ in. to the ft.	⅛ in. to the ft.
Stone block	½ in. to the ft.	¼ in. to the ft.
Wood block	¼ in. to the ft.	⅛ in. to the ft.

foundation is to be constructed on the sub-grade, proper care should be taken in its preparation in order that the greatest economy may be had in the design and expense for the artificial foundation, and, generally speaking, at least, the higher the type and the more expensive the artificial foundation, the greater care should be had to develop to the utmost the possibilities of the sub-grade. Uniformity in the composition and compaction, as well as evenness of its surface, is far more important than has been generally considered necessary, and permanence of all the desirable qualities in the sub-grade is equally important.

When an artificial foundation or a cement-concrete pavement is used, the sub-grade should be as carefully prepared, rolled, and compacted, as for any other roadway surfacing, and should be made to conform accurately to the proper lines and grades. If necessary, the sustaining powers of the natural foundation should be reinforced and the strains in it thereby further distributed by the interposition between it and the cement-concrete slab of an artificial foundation consisting of a layer, or layers, of sand, gravel, broken stone, or similar material. Reliance should not be placed on the concrete, if used, for bridging soft, spongy, or unyielding spots; all vegetable or perishable matter should be removed from the sub-grade, and such other material substituted as will insure a thoroughly compacted and homogeneous sub-grade for the concrete. It may be necessary, for proper compaction, to use water in connection with rolling, and, in any event, cement-concrete should not be deposited on a dry, absorbent sub-grade.

Every precaution should be taken to prevent a disturbance of the sub-grade after it is completed and until the next layer is deposited.

When a cement-concrete or other rigid pavement is to be constructed over an old surfacing composed of gravel or broken stone macadam, the old pavement should be loosened, spread to the full width of the new pavement, and then thoroughly compacted, filling the interstices with fine material and re-rolling until a dense sub-grade is obtained.

Artificial Foundations.—Where the character of the traffic justifies the use of an artificial surfacing, it also demands a correspondingly strong foundation. Whether or not an artificial foundation shall be supplied will depend on the local conditions, but, in the selection of the materials and the methods of construction of the artificial foundation, every consideration should first be given to the possibilities for securing the greatest efficiency from the natural foundation. Economy in reference to the roadway will be had from the proper choice of the various materials available for artificial foundations, such as sand, gravel, broken stone, and concrete.

Owing to the inherent lack of elasticity in brick or cement-concrete pavements, it is especially necessary that the surface of the wearing course of such pavements shall be built and remain smooth and true to contour, for the sake of ease of traction, comfortable riding, and integrity of the surface, particularly where a cement filler for the joints is used. Special care, therefore, should be taken in these cases to provide foundations of ample strength and permanence to this end. Stone block pavements should generally, and wood block pavements should always, be laid on a cement-concrete foundation, but, in case of temporary paving with stone blocks, any type of stable foundation may be used.

Local conditions, occasionally, may justify the omission of an artificial foundation for brick pavements where the natural foundation material can be satisfactorily prepared

and a reasonable permanence then expected from it, where a relatively low first cost of the brick prevails and where light traffic only is to be expected.

Any artificial foundation should be of a substantial thickness and be properly consolidated; and should be rendered so compact that further settlement or displacement will be avoided to the utmost.

The most usual proportions for a cement-concrete foundation have been one part cement, three parts fine aggregate, and six parts coarse aggregate. This standard, however, is empirical rather than scientific, and a more rational proportion should be developed according to the needs and facilities of each case. Sometimes it may be desirable to increase the mass at the expense of unit strength, or to increase the mass for the sake of economy in the more expensive material. In mixing, placing, and completing a cement-concrete foundation, the principles expressed under the head of "Cement-Concrete Pavements" apply, and reference thereto should be had.

Materials.—Having determined the characteristics desirable for the materials to be used in any highway work, their description in the specifications should be concise, clear, and exact. Although, in some cases, it may not be possible to designate precisely the characteristics desired, it will be possible to specify that these qualities shall lie between certain limits, thus giving a reasonable tolerance to the determination of the quality by test as well as avoiding uncertainty as to whether or not a quality in this respect of a material offered is suitable. The description of a material by means of a trade name is permissible only in most unusual cases, and such a description as "equal to" another similar material should never be used. Qualities of a material or methods of its use should not be left "to the satisfaction of the engineer" or "as determined by, or in the opinion of, the engineer."

Specific tests and such description of the methods of performing each test as will leave no room for doubt as to whether the results of the tests come within the limits of tolerance should always be expressed, either in detail or by reference to the standards of some reputable authorities. The tests and methods of performing them, to be found in detail in Appendices B and C, are recommended for this purpose.

Whenever comprehensive specifications are to be prepared so as to admit a variety of types of bituminous materials, separate specifications, as may be necessary, should be written for each type.

Joints.—For the ordinary joints in block pavements, the materials and methods of filling should be selected so as to produce, not only a surface which will retain to the utmost its imperviousness and the stability of the blocks themselves in place, but, also, they should, as far as practicable, conduce toward evenness of wear of the surface of the pavement. If the blocks are resistant to abrasion, but are inclined to round off at the edges of the upper surface under traffic, such filling of the joints is desirable as will reduce rounding off at the joints. On steep grades, where some roughness of surface may be desirable for the sake of affording better foothold for animals, some openness at the top of the joint is desirable, and the softer joint fillers, less resistant to wear, may be preferred.

Joint fillers naturally are divided into two main classes—the hard cement-mortar filler and the soft bituminous filler. As it is desirable to secure water-proof pavements, sand alone should never be used as a joint filler. Though the use of sand joints may occasionally appear to be justified in the interests of economy, it will generally be found unwise to use such joints when some relatively slight additional first cost will result in appreciably prolonging the life of the pavement. A bituminous filler may be preferred to a cement-grout filler on account of the lower cost of street opening repairs, the better foothold provided for horses, and the securing of a more elastic and less noisy pavement.

Cement-mortar joints when properly made will conduce to integrity of the surface. The proportions of sand, cement, and water will be affected by local conditions. To insure the best results, a 1:1 mix of sand and cement is recommended. Great care is necessary in mixing and applying the mortar or grout. Uniformity in the cement grout, and especially skill and care in its application, are essential to success. To insure uniformity, there should be a constant agitation of the mix, up to the moment of its application, and no more water than is necessary for proper fluidity should be used. Ample time should always be allowed for the grout to set thoroughly before the traffic is admitted to the roadway.

With bituminous joint fillers, care must be taken to select materials which will not be too brittle in cold weather and so chip out from the joints under traffic, and which will not be so soft in hot weather as to flow out of the joints between the blocks. It is believed, although not yet generally admitted as having been actually proved by experience, that

the use of a bituminous mastic for joint filling would be an improvement over the customary practice of using bituminous material alone for this purpose. One of the great difficulties with bituminous fillers of any kind will be that of properly filling the joints between the blocks, and great care must be taken to insure this result.

Expansion-contraction Joints.—Joints at intervals across certain types of pavements, such as brick and cement-concrete, as well as along the curbs, have been used in order to compensate for a more or less unavoidable movement of the pavement slab, which takes place under different conditions of moisture, or temperature of the air. In cases where expansion-contraction joints across the roadway at intervals are decided on, the Committee recommends the use of bituminous material and the abandonment of all forms of the so-called "armored" joints, because of the smaller interruption to the homogeneity of the roadway surface thus secured.

Cushion Courses.—The function of a cushion between the brick or block on an artificial foundation of cement-concrete is to allow for irregularities in the surface of the concrete and in the depths of the brick or block, and to give resiliency to the wearing course. If the surface of the concrete foundation is made true to the adopted cross-section, and as the variation in depths of the brick or block decreases, the thickness of a sand cushion may be correspondingly decreased. The thickness should never be greater than that necessary to compensate for the unevennesses referred to plus such a thickness in the layer as will enable the latter to perform satisfactorily its function as a cushion, that is to say, about ½ in. for this latter addition. If the surface of the concrete is truly parallel with the finished pavement, and if the variation in the depths of the bricks or blocks does not exceed ⅛ in., the thickness of a sand cushion can safely be reduced to ¾ in. The material which has generally been used for the cushion course is sand, but engineers, recently, have been considering the advisability of using Portland cement mortar instead, the mortar being spread either on the surface of the concrete foundation after the latter has been completed, or being laid as a part of the foundation itself. The objection made by some to mortar is that it makes too solid a base for the blocks and gives no resiliency. This at present seems to be a moot question. The substitution of the mortar results in a monolithic structure, perhaps of greater strength, but less capable of absorbing shock without injury than is the case where a sand or bituminous cushion is introduced between the wearing surface and the foundation, especially where the joints are filled with a bituminous filler.

The sand for a cushion course may be slightly more loamy than that permissible for safety in mortar or cement concrete work. This excess loaminess may be advantageous in offering the sand a greater ability to resist displacement; but it should not be so loamy or fine as to prevent proper spreading and compaction, or to afford such a degree of capillarity as will result in frost action.

A bituminous mixture composed of sand, stone screenings, or possibly wood fiber, and a bituminous cement would probably be more satisfactory for many reasons than either the mortar course or the sand alone, and such a bituminous mixture needs to be only from ¼ to ½ in. in thickness, provided the surface of the concrete foundation is made sufficiently smooth and regular in contour.

Finishing of Surface.—An objectionable slipperiness of many pavements may be decreased or prevented by proper precautions during construction or by proper treatment thereafter. The length of time that a finished pavement should be closed to traffic in order to season properly before use varies from a few hours to several days, dependent on the character of the material and methods used and on climatic and other local conditions. Pavements in which Portland cement is used for filling the joints or in the mass of the surfacing itself should seldom, if ever, be closed for less than 2 weeks after completion. Although the plans and specifications usually call for the surface to be finished to definite cross-sections, grades, or contours shown on the plans, questions frequently arise under contracts as to the importance of variations in exactness of compliance in the finished surface.

The Committee has had a number of observations made, from the results of which it is convinced that, in a newly completed pavement, the variations from a straight-edge or template, 8 ft. in length, should not exceed ⅛ in. for asphalt block, bituminous concrete, brick, cement-concrete, sheet-asphalt, and wood block pavements, and ¼ in. for broken stone roads, and bituminous macadam and stone block pavements.

Manhole Covers and Street-car Tracks in Roadway Surfaces.—Uniformity in the roadway surface being essential for minimum wear and expense of maintenance, anything, such as manhole covers or street-car tracks, introducing an element of non-uniformity into the surface, should be counteracted as far as possible whenever surfaces of different degrees of

hardness adjoin in a pavement. The traffic coming from the harder to the softer surface naturally causes abnormal wear on the latter.

Practically all manhole covers are laid on rigid masonry, so that, unless some special treatment is given to the pavement, there is apt to be undue wear adjacent to such structures. Where the pavement is of stone block, wood block, or brick, the difference in hardness between them and the manhole heads is not so great as with other paving materials, so that, when these pavements are used, they can be laid flush with the manhole covers, provided extra care is taken with the foundation immediately adjacent to the heads, to prevent any possible settlement. In the case of macadam or bituminous pavements, a different treatment should be used. In such cases the pavement should be laid about $\frac{1}{8}$ in. above the manhole head. This will prevent the abnormal wear caused by the pounding action of the wheels of vehicles, which would occur if the pavement surface was any distance below that of the manhole head, a condition which must exist if the pavement is laid level with such heads, as it is not possible to compact the pavement so that it will not compress under the traffic, forming depressions before those actually caused by wear begin.

In the case of car tracks in streets, modern construction is such that the tracks are nearly rigid, although this condition does not exist in all cities at present. Where it does exist, however, the pavement should be laid, as in the case of manhole heads, somewhat above the level of the rail. The Committee, however, does not believe that in any case a bituminous pavement should be laid between the tracks or between the rails of the tracks. When car tracks are laid in roads the construction is not generally as nearly rigid as in streets, and the rails are usually of the T form. The tracks in such cases are often laid at the side of the road, rather than in the center, as is customary in streets. In the case of macadam or bituminous roadways, and when the rails are in the center, it would be advisable to lay stone blocks or brick for a width of at least 18 in. adjacent to the rails; when the rails are at the side and the railroad area does not form a part of the roadway proper, loose broken stone or gravel may be substituted for the stone blocks or brick.

ASPHALT BLOCK PAVEMENTS[1]

General.—Specifications for asphalt block pavement should cover thoroughly the several components of the bituminous concrete used, the manufacture of the blocks, the blocks *per se*, and the details of construction of the pavement.

Materials.—Experience has demonstrated that the blocks should be composed of asphalt cement, crushed trap rock or equally hard and tough material, and mineral dust. All particles of the trap rock should pass a $\frac{1}{4}$-in. screen, and the mineral dust or filler should consist of powdered limestone. The bitumen content of the blocks should be between 6.5 and 8.5 per cent., depending on the grading of the mineral aggregate and the method of manufacture. The specifications should contain specific requirements with reference to the asphalt cement, filler, and the grading of the mineral aggregate, which latter should be similar to the following:

Passing 200-mesh sieve	20 to 35 per cent.
Passing 80-mesh sieve and retained on 200-mesh sieve	7 to 15 per cent.
Passing 20-mesh sieve and retained on 80-mesh sieve	12 to 30 per cent.
Passing $\frac{1}{4}$-in. screen and retained on 20-mesh sieve	30 to 50 per cent.
Retained on $\frac{1}{4}$-in. screen	0 per cent.

The specifications should also cover the specific gravity of dry blocks, which should not be less than 2.45 at 25°C. (77°F.) and the percentage of absorption of water of the blocks, after being dried for 24 hours at a temperature of 65°C. (149°F.), should not be more than 1 per cent. after immersion in water for 7 days. The blocks should be about 5 in. in width and 12 in. in length, and 2, $2\frac{1}{2}$, or 3 in. in depth, depending on traffic conditions.

Construction.—The blocks should be laid on a fresh $\frac{1}{2}$-in. mortar bed which covers a cement-concrete foundation. After being laid, the blocks should be covered with a thin layer of clean, dry, fine, sharp sand which should be thoroughly swept into the joints until they are filled.

BITUMINOUS CONCRETE PAVEMENTS[1]

Classification.—The principles to be covered in drafting specifications for bituminous concrete pavements will be grouped under the three classes into which these pavements generally may be divided. These classes are described as follows:

[1] For discussion of rates of grade, crown, artificial foundation, etc., see "General Conclusions, page 439."

Class *A*.—A bituminous concrete pavement having a mineral aggregate composed of one product of a crushing or screening plant;

Class *B*.—A bituminous concrete pavement having a mineral aggregate composed of a certain number of parts by weight or volume of one product of a crushing or screening plant and a certain number of parts by weight or volume of sand, broken stone screenings, or similar material, with or without a filler;

Class *C*.—A bituminous concrete pavement having a predetermined, mechanically graded aggregate composed of broken stone, broken slag, gravel, or shell, with or without sand, Portland cement, fine inert material, or combinations thereof.

BITUMINOUS CONCRETE PAVEMENTS, CLASS A

Mineral Aggregate.—Broken stone, because of the satisfactory bond secured, should be used wherever possible, although bituminous concretes constructed with gravel have proved satisfactory for light traffic where great care has been taken in the selection of the gravel and in the construction of the pavement.

Broken stone should be clean, rough-surfaced, sharp-angled, of compact texture, and uniform grain. If the pavement is to be subjected to medium or heavy traffic, the broken stone used for the construction of the wearing course should show a loss or abrasion of not more than 3.5 per cent., and its toughness should not be less than 13.

Especial care is required in drafting the specifications covering the broken stone or gravel to be used. An excess of large or small sized stone or gravel should be avoided. Practice has demonstrated that a mineral aggregate composed of those materials which will comply with the following mechanical analysis, using laboratory screens having circular openings, will produce satisfactory results: All the material shall pass a 1¼-in. screen; not more than 10% nor less than 1% shall be retained on a 1-in. screen; not more than 10% nor less than 3% shall pass a ¼-in. screen.

Bituminous Cements.—Experience has demonstrated that the most efficacious bituimnous concrete pavements of Class *A* are constructed by using suitable asphalt cements or refined tars in the mix, and asphalt cements for seal coats. Satisfactory results will be secured when tar cements are used for seal coats, but the surface must be re-treated more frequently than when asphalt cements are used.

Heating Aggregates and Bituminous Cements.— Although satisfactory pavements have been constructed using unheated mineral aggregates and suitable bituminous cements, service tests demonstrate that the best results are secured by using for the mineral aggregate broken stone or gravel which is heated until thoroughly dry to between 66°C. (150°F.) and 121°C. (250°F.). If revolving dryers in which the flame is permitted to come in contact with the aggregate are used, great care should be taken to ensure uniformity of heating and so avoid the danger of burning the aggregate.

In order to obtain a fluidity of the bituminous material which should be sufficient to ensure a proper coating of the mineral particles in cases where a heated aggregate is used, and also to prevent injury to the bituminous material, the asphalt cements should be heated to a temperature between 135°C. (275°F.) and 177°C. (350°F.), and refined tars to a temperature between 93°C. (200°F.) and 135°C. (275°F.).

Mixing.—The quantity of bituminous cement to be used in the mix will depend on the kind of broken stone or gravel and bituminous cement, the character of the aggregate, the climatic conditions, etc. For the aggregate heretofore mentioned, the bituminous concrete mixture should contain between 5 and 8 per cent. by weight of bitumen.

The bituminous concrete should be mixed in mixers designed and operated so as to produce and discharge a thoroughly coated and uniform mixture of non-segregated aggregate and bituminous cement. Except on small contracts and for repair work, mixers which provide for the heating of the aggregate by the use of a flame in the mixing chamber should not be used, on account of the danger of burning the aggregate or the bituminous cement.

Laying.—To ensure ease of manipulation and the proper compaction of the bituminous concrete, the mixture as delivered on the roadway should have a temperature of not less than 66°C. (150°F.). Experience has demonstrated that a thickness, after rolling, of 2 in. of bituminous concrete is economical and efficacious. In order to secure an even surface and adequate compaction by a thorough interlocking of the particles of the aggregate, a tandem roller weighing between 10 and 12 tons should be used.

Seal Coat.—A seal coat should always be used on this type of bituminous concrete, as maintenance charges and annual cost will be reduced materially thereby. The seal coat

should consist of from ½ to 1 gal. per sq. yd. of bituminous cement, uniformly distributed, preferably by the use of a hand-drawn distributor followed by a squeegee. The bituminous cement should be covered with an application of dry stone chips, which should be rolled.

Seasonal Limitations.—Bituminous concrete of this type should not be mixed or laid when the air temperature in the shade is lower than 10°C. (50°F.), as otherwise it is difficult, under average conditions, to secure an even and well compacted wearing course.

BITUMINOUS CONCRETE PAVEMENTS, CLASS B

Specifications for pavements of this class have generally stipulated that so many parts of broken stone or gravel and so many parts of sand or other fine material are to be mixed with a certain quantity of bituminous cement. By the use of this specification, and with unusual supervision, it is practicable to secure a fairly well-graded aggregate, but in most cases the mixture will be found to contain an excess of broken stone, with insufficient fine material to fill the voids therein, and in other cases it will contain an excess of sand in which the broken stone is held as isolated particles.

BITUMINOUS CONCRETE PAVEMENTS, CLASS C

This type includes the so-called "Topeka" mixture, and several kinds of patented pavements.

Topeka Bituminous Concrete Pavement.—If the Topeka pavement specification embodies the grading, as contained in the decree of 1910, namely,

Bitumen, from 7 to 11 per cent.
Mineral aggregate, passing 200-mesh screen, from 5 to 11 per cent.
Mineral aggregate, passing 40-mesh screen, from 18 to 30 per cent.
Mineral aggregate, passing 10-mesh screen, from 25 to 35 per cent.
Mineral aggregate, passing 4-mesh screen, from 8 to 22 per cent.
Mineral aggregate, passing 2-mesh screen, less than 10 per cent.,

special provisions should be made in the specifications covering the broken stone and sand to be used, in order to secure satisfactory grading. Otherwise, the principles stated under "Bituminous Concrete Pavements, Class *A*," should be followed, except that a seal coat is not considered necessary under many conditions where this type of pavement is used.

Patented Bituminous Concrete Pavements.—In cases where patented bituminous concrete pavements of Class *B* or Class *C* are used, the sam fundamental principles observed under "Bituminous Concrete Pavements, Class *A*" should be followed, especially in the case of covering in detail the composition and grading of the mineral aggregate, and the physical and chemical properties of the bituminous cements used.

BITUMINOUS MACADAM PAVEMENTS[1]

Materials.—The broken stone should be of a quality equal to that prescribed for broken stone roads, and should have the same characteristics. The bituminous materials may be of asphalt or refined tar.

Construction.—The principles relating to thickness applicable to a broken stone road are likewise applicable to bituminous macadam pavements, and thorough rolling, including the rolling of the upper course, both before and after the application of the bituminous material, is also necessary. As it is desired to bind only the upper course with bituminous material, it is necessary, in order to prevent waste by penetration, that there should be no appreciable voids in the next lower course. It is not necessary, however, to flush the filler or binder in this course to the same extent as is necessary in binding the top course of a water-bound road, and it is absolutely essential that no binder should cover the stones of the lower course when the top course is spread.

The quantity of bituminous material used should be only sufficient to penetrate through the upper course. The quantity per square yard cannot be prescribed absolutely, depending in some degree on the hardness and size of stone used, but, in general, the application of 1 gal. or less to the square yard for each inch in thickness of the finished upper course is adequate.

The use of a pressure distributor in applying the bituminous material is essential, and

[1] For discussion of rates of grade, crown, artificial foundation, etc., see "General Conclusions, page 439."

the distributor should be of such type that absolutely uniform application may be accomplished, and that no ruts are formed in the surface by the wheels supporting the distributor.

The bituminous material should be applied at such a temperature that it will flow freely, and, to insure proper penetration, the stone should be dry and clean, and the air temperature should not be lower than 10°C. (50°F.) during application.

In order to secure a proper surface, the covering material should preferably consist of the crusher product passing over a ¼-in. screen and through a ¾-in. screen. Finer material, however, may be used for covering if a slippery surface is not objectionable, but the use of material passing through a 10-mesh sieve should be avoided.

The use of a bituminous material by no means justifies any lack of care in the ordinary details to be followed, but rather increases the need for thoroughness and skilled supervision. The main principles underlying good construction in water-bound roads remain in full force when such roads are treated with bituminous material. Whatever method may be used in any case, it is as essential, in bituminous work as in water-bound construction, that a suitable quality of road metal be used.

BITUMINOUS SURFACE TREATMENTS[1]

Description.—The proper treatment of a broken stone, gravel, shell, or slag roadway with bituminous material, for the purpose of eliminating the so-called dust nuisance, will at the same time render even the best of such roadways more efficient for sustaining traffic, and such treatment with bituminous materials is usually preferable and more economical than sprinkling with water, or the use of hygroscopic salts.

Bituminous Material.—Either refined tar, cut-back asphalt, or asphaltic oil may be used for surface treatment. If the surface to be treated is a gravel, broken stone, slag, or other porous and non-bituminous crust, practice has proven that bituminous material of such consistency that it can be applied at a temperature below 52°C. (125°F.) is preferable to heavier material, and that on any crust the application of a quantity in excess of ½ gal. per sq. yd. is inadvisable. It is advisable to apply the material in quantities not exceeding ¼ gal. per sq. yd. at a time. Heavier material in less quantity, however, should be used on bituminous roadways; otherwise there will be a tendency toward an objectionable softening of the material previously used in the construction of the roadway.

Construction.—The surface to which a bituminous treatment is to be applied should be dry, compact, and free from depressions and dust. On a broken stone road, the application should be made on the exposed stone surface of the upper course, such exposed surface being obtained by thoroughly removing with brooms or sweepers the binding material or dust that may have been applied or accumulated thereon. The bituminous material, in all cases, should be applied by a pressure distributor designed so that the material will be spread uniformly and with a pressure of not less than 20 or more than 75 lb. per sq. in.

The application, in all cases, should be carried over the outside edges of the rolled metal and on the shoulder far enough to protect the edges of the metaled surface. The material should be applied only after the surface has been thoroughly compacted by traffic or otherwise.

After the bituminous material is applied it should be covered with the toughest grit obtainable, preferably of a size that will pass through a screen having openings of not less than ⅜ in. nor greater than ⅝ in., just enough of such material being used to cover the bituminous material. It is advantageous, but not entirely necessary, to roll with a steam roller after the application of the grit.

BRICK PAVEMENTS[1]

Cushion Course.—In the case of brick pavements, the brick being of uniform size, sufficient resiliency will be secured by the use of a sand cushion 1 in., or even slightly less, in depth, provided the depth is uniform and the surface of the concrete foundation is truly parallel with the finished pavement; and if the variation in the depth of the brick does not exceed ⅛ in., the thickness of the sand cushion can safely be reduced to ¾ in. In many brick pavements recently laid the cushion course has been dispensed with entirely, the brick having been bedded in cement mortar spread over the concrete foundation. This results

[1] For discussion of rates of grade, crown, artificial foundation, etc., see "General Conclusions, page 439."

in a monolithic structure less capable of absorbing shock than is the case where a sand or bituminous cushion is introduced between the wearing surface and the foundation. A cushion course composed of sand or stone chips and a bituminous cement about ½ in. in thickness may be substituted for sand or cement mortar, provided the surface of the concrete foundation is made sufficiently smooth and regular in contour.

Materials.—The quality of the brick should be determined by physical tests. The method of making the rattler test adopted by the American Society for Testing Materials is approved by the Committee. This test will indicate the toughness and resistance to wear from shock and abrasion. Uniformity in the rate of wear is so important that it properly may be a controlling consideration, even at the expense of a moderate increase in the rate of wear. In size and shape it is desirable to conform to accepted standards in order that repairs and renewals may more readily be made. Uniformity in size is especially important, and variations in depth should be kept within the narrowest limits.

Construction.—The bricks should be laid in straight courses at right angles to the axis of the roadway, although at intersections they may advantageously be laid in diagonal courses arranged so that traffic turning any of the corners will move across the bricks and not along the continuous joints. They should be laid so that the joints will be uniform in width and of sufficient width only to permit a filler to reach the bottom of the joints. Lug bricks have the advantage of insuring such uniform width with ordinary care in laying. If a sand cushion is used, great care should be taken to avoid any disturbance of the surface of the cushion after it has been brought to true grade by using a template. If bedded in a mortar or bituminous cushion, the brick should be bedded so that the surface will be as true as possible. In all cases the brick after being laid should be brought to a true and even surface by the use of a roller.

BROKEN STONE ROADS[1]

Materials.—All broken stone should be clean, rough-surfaced, angular, of compact texture, and of uniform grain. It should preferably be of such quality that, using standard laboratory tests, it will show a percentage of wear not greater than 5 (French coefficient of wear not less than 8), and a toughness of not less than 6.

The broken stone should be separated into component sizes by passing the product of the crusher over rotary screens having circular openings, the different sizes of stone being collected in separate bins. The separation of sizes is governed somewhat by the thickness at which the crust is to be laid. If a 6-in. surface is to be laid, the maximum size should not exceed that of stone passing a 3-in. screen, whereas, if the pavement is to be 7 in. or more in thickness, a 3½-in. screen is permissible. In all cases the stone should be spread and rolled in two or more courses, the largest size generally being used in the lower course and the smaller sizes in the upper course or courses, each course being spread in such manner that there will be uniformity in its density. Such uniformity is accomplished by spreading from piles dumped on boards or alongside the road, or by automatic spreading wagons.

The product passing through a ⅜ to ¾-in. screen should be used as a binder, and on two-course work should be applied on the top course only, whereas, on the three-course work, the second course should be lightly covered with this material.

Products of broken stone obtained from portable and stationary stone-crushing and screenings plants, though complying with a given specification of the type now ordinarily used, vary to a considerable extent, due to variations in the plant and its operation, such as kind of rock, type and method of operation of crushing plants, methods of separation of products, differences in lengths and diameters of sections of rotary and shaker screens, and differences in inclination and rate of operation of screens.

The necessity for more carefully drawn specifications covering the sizes of the particles of which a given product of a stone-crushing and screening plant is composed, is illustrated by Table 3, mechanical analyses of two products obtained from the same plant, both of which products passed over a section of a rotary screen having circular holes 1¼ in., and through a section of a rotary screen having circular holes 2¼ in. in diameter.

It is obvious that, for many forms of construction, in order to secure successful results greater care must be used in the writing of specifications for products of broken stone. The Committee recommends the general use, as soon as practicable, of the "Standard Form of Specifications for Certain Commercial Grades of Broken Stone," as recommended by Committee D-4, of the American Society for Testing Materials, in its 1916 Report.

[1] For discussion of rates of grade, crown, artificial foundation, etc., see "General Conclusions, page 439."

TABLE 3

	Sample "A," percentage	Sample "B," percentage
Passing ¼-in. screen	0.3	0.2
Passing ½-in. screen and retained on ¼-in. screen	0.4	1.1
Passing ¾-in. screen and retained on ½-in. screen	2.2	12.6
Passing 1-in. screen and retained on ¾-in. screen	8.0	37.5
Passing 1¼-in. screen and retained on 1-in. screen	29.1	40.9
Passing 1½-in. screen and retained on 1¼-in. screen	27.1	7.7
Passing 2-in. screen and retained on 1½-in. screen	32.9	0.0
	100.0	100.0

The broken stone shall consist of one product of the operation of a stone-crushing and screening plant, without re-combining or mixing, and shall conform to the following mechanical analysis, using laboratory screens:

Passing ..... in. screen (having smallest holes selected) from ..... to ..... per cent.
Passing ..... in. screen (having next to largest holes selected) from ..... to ..... per cent.
Passing ..... in. screen (having largest holes selected) from ..... to ..... per cent.

In this form of specification an attempt is made to cover in the mechanical analysis only the limits of the smallest and largest particles. No attempt is made to secure a carefully graded aggregate, but simply a product suitable for the type of road or pavement in question.

An engineer should base the selection of screens, to be used in the specification for a given product of broken stone, on the results of mechanical analyses of many similar products obtained from portable and stationary crushing and screening plants which supply the locality in which the specification is to be used.

Construction.—Each course of a broken stone road should be thoroughly rolled with a roller weighing from 10 to 15 tons, the rolling being done first along the sides and gradually approaching the center, and being continued until there is no movement of the stone ahead of the wheels of the roller.

The binder should be used on the top course in such quantity that, after alternate spreading of binder and watering, with continuous rolling, the voids become so filled as to result in a wave of grout being pushed along the surface by the front wheel of the roller.

After the completion and binding of the top course, a thin layer of screenings or stone dust should be applied to the surface in sufficient quantity to cover it evenly.

CEMENT-CONCRETE PAVEMENTS[1]

General.—A thickness of from 5 to 8 in., as stated in the general principles, may ordinarily be considered sufficient for a concrete slab, and, if it seems advisable, from motives of economy, the thickness may be diminished from the center of the slab to the edges. Special conditions may call for variations, even outside of the limits given. The character and the drainage of the sub-grade, its probable stability as a foundation, as well as the nature and amount of traffic, are some of the factors which enter into the rational determination of the thickness of the slab.

Materials.—The cement should be tested by the methods recommended by the Special Committee on Uniform Tests for Cement[2] of the American Society of Civil Engineers, and

[1] For discussion of rates of grade, crown, artificial foundation, joints, etc., see "General Conclusions, page 439."

[2] *Trans. Am. Soc. Civ. Eng.*, **75**, (1912), p. 665.

should meet the requirements adopted by the American Society for Testing Materials, as printed in the 1916 Year Book of that Society.

The Committee wishes to emphasize the importance of the aggregate in making up the concrete structure. Fine aggregate may be considered as gravel, sand, or screenings from hard, durable rock, graded so that, when dry, it will pass a screen having ¼-in. circular openings. The best fine aggregate is in general that which is graded fairly uniformly from the ¼-in. size mentioned above, downward, but not more than 5 per cent. should be of such fineness that it will pass a sieve having 100 meshes per linear inch. A preponderance of the coarser particles rather than the finer is desirable, and, in any case, less than 3 per cent. of the material should pass the 200-mesh sieve. Standard briquettes, made of samples of fine aggregate and tested at the usual periods of 7 and 28 days, should show a strength at least equal to similar briquettes made of the same cement and three parts of standard Ottawa sand. Coarse aggregate, in general, should not be larger than will pass a screen with 2-in. circular openings, ranging down fairly uniformly from this size to that retained on a ¼-in. screen. The loss of such material, as determined by the abrasion test, should not be more than 5 per cent.

A denser and more uniform concrete may be made by screening the material, both fine and coarse aggregates, into different sizes and re-combining these different sizes in such a way as will give the densest mixture, that is, the smallest percentage of voids, when dry. Although this adds somewhat to the expense, the resulting composition will generally be found enough better to make up for the additional expenditure. Furthermore, in proportioning the ingredients of the concrete, a mixture based on mechanical analyses is in general to be preferred to the arbitrary rule of a 1:2:4 or 1:3:5 mix. The slight increase in time and expense which is incurred by determining the voids and combining the various sizes to get the greatest density is more than repaid by the strength and density of the resulting mixture. Similar care in the determination of the proper quantity of water is also to be desired in order that the concrete may have a uniform consistency as it is deposited in place. Though the mixture should be rather wet, especially if it is deposited without tamping, it should still be stiff enough to hold its shape when struck off by the template, and yet not result in segregation of the different sizes throughout the mass. Water used in mixing should be clean and free from oil, alkali, or vegetable matter.

Construction.—Forms used in cement-concrete pavements should be as carefully considered as those for any other class of structural work in concrete. It is important they be true, and free from warp, and of sufficient strength to hold the wet concrete without springing out of shape. Particular care should be taken to keep the forms tight, so that leakage through the sides, which might allow the cement or mortar to be carried out of the coarse aggregate along the edges of the roadway, may be effectively prevented. The concrete should be deposited rapidly on the sub-grade to the required depth and to the entire width of the pavement. It is better to have the surface of the rolled and finished sub-grade thoroughly dampened before beginning to deposit the concrete. Rolling or ramming the freshly placed concrete is desirable wherever practicable, as it not only increases the density of the resulting mass, but also tends to place the particles of the coarse aggregate on the surface, so that they interlock with each other and present a flat side to the wear of traffic.

Clean vertical joints, straight across the roadway, through the entire mass of concrete in place, should be insisted on when the work stops for a day, or if there is a stoppage of more than 30 min. in the work during the day. Special precautions should be taken to prevent freezing when work is carried on during cold weather, and it should be borne in mind that concrete sets much more slowly in cold weather than in warm weather. If, in the course of the work, the temperature reaches, say, 40°F., and is falling, the operation of mixing and laying concrete should be suspended, and the newly laid surface suitably protected from frost.

In finishing the surface of the concrete, a template or a striking board should be used, which gives the true form of the finished pavement for its entire width. For the final surface finish, the use of the wooden float, operated from a suitable bridge which spans the entire pavement, is desirable, although satisfactory results may be obtained by the use of a properly operated belt about 10 in. wide. Care should be taken that the final surface of the pavement is true, both transversely and longitudinally, that is, with regard to both cross-section and grade. In all cases the surface of the finished pavement should be kept wet and protected from the sun for several days.

EARTH AND SAND-CLAY ROADS[1]

Materials for Earth Roads.—The earth road should be constructed of the natural soil, from which, for a depth of 10 in. at the center to 5 in. at the sides of the roadway, all stones more than 3 in. in diameter and all sods, roots, and similar materials should be removed.

Construction of Earth Roads.—The soil should be mixed with plows, disk and spike-tooth harrows, or by other satisfactory means, until all the surfacing material is of uniform texture. When the earth roadway is completed its thickness should be not less than 10 in. at the center and 5 in. at the sides. The roadway should be shaped by the use of a road grader, and should be finished so as to conform to the desired cross-section, the crown of which should usually be about 1 in. to the foot. If any depressions appear during the grading of the roadway, they should be carefully filled with soil and the roadway reshaped. In order to secure satisfactory results, several months should elapse after the road is graded before it is considered complete; and such settlements and irregularities as develop should be corrected by the use of a grading machine or road drag.

Materials for Sand-clay Roads.—Top-soil or mixtures of sand and clay should comply with the following requirements: The sand content should be at least from 70 to 80 per cent.; the sand used in sand-clay mixtures should preferably be composed of hard angular particles at least 30 per cent. of which should be retained on a 50-mesh sieve; the clay content should vary from 10 to 20 per cent and under no circumstances should be allowed to exceed 30 per cent.; the clay, when placed on the roadway, should not contain lumps larger than 3 in. in diameter.

Construction of Natural Sand-clay Roads.—The top-soil should be spread on a flat subgrade to a depth of 10 or 12 in. at the center and 5 or 6 in. at the sides. After the surfacing material has been laid, the ditches should be constructed, and the material obtained therefrom should be used in constructing shoulders. It is advisable to plow and then harrow the surfacing material, as a more homogeneous layer is secured thereby. The surfacing material should not be compacted with an ordinary roller but with a sheep-foot roller, or the compaction should be obtained with the hoofs of animals and the wheels of vehicles going over the roadway. For successful results, thorough puddling is necessary, and this can practically only be secured through the medium of rains during the period of compaction. During compaction and after each rain the surface of the roadway should be immediately reshaped and crowned. When completed, the compacted roadway should have a depth of from 7 to 8 in. in the center and 3½ to 4 in. at the edges, and have a crown of about ½ in. per foot.

Construction of Sand-clay Roads on Clay Sub-soil.—Although there are several methods of construction, the following is advocated, as uniformly satisfactory results are secured by its use. The roadbed should be graded practically level, and the portions to be constructed of sand-clay should be excavated to a sufficient depth so that the earth removed and placed on the shoulder of the road will give the right slope to the ditches. The depth of excavation should be from 1½ to 3 in., depending on the width of the road between ditches. The clay soil should then be plowed to a depth of 2 or 3 in., after which about 4 in. of sand should be spread evenly over the surface and thoroughly worked in with a disk harrow. Then 4 in. more of sand should be added and harrowed. During a rain, the sand-clay mixture should be thoroughly harrowed, and after the rain the roadway should be dragged into shape.

Construction of Sand-Clay Roads on Sand Sub-soil.—On a practically flat roadbed, a layer of clay from 2 to 4 in. in thickness should be spread evenly over the roadway. A layer of clean sand should then be spread over the clay, and the roadway thoroughly harrowed. The roadway should be shaped up with a drag, and during a rain it should be again harrowed. The roadway should be finally shaped with the use of a grading machine or drag.

GRAVEL ROADS[1]

Description.—The subject of gravel roads embraces a great variety of styles of construction, from the simple expedient of surfacing the existing roadway with run-of-the-bank gravel from near-by pits to the construction of an improved highway with all the necessary drainage structures and improvements of grade attending a broken stone roadway.

[1] For discussion of rates of grade, crown, artificial foundation, etc., see "General Conclusions," page 439.

Materials and Construction.—The method of treating the gravel itself may vary from the application as it is found in the pit to the separation into sizes, and even to passing the gravel through the crusher before screening. In general, we may separate gravel roadways into two classes:

First, those in which the gravel is screened and applied in the same manner as with a broken stone roadway. This may be with or without crushing the gravel.

Second, those in which the gravel is applied to the roadway in its natural state as found in the pit, with or without the addition of other material, or the natural material may be passed through the crusher before application to the roadway.

In the first case, with rounded gravel, the tendency toward dislodgment under traffic is greater than with angular broken stone, and hence, in order to reduce this tendency, the size of the pieces in the courses of the roadway must be somewhat smaller with gravel than in the case of broken stone.

In the second case, the selection of the material will be governed by the gravel available in the locality where the roadway is to be constructed. Every endeavor should be made to select a material that will show, by tests, its fitness as regards hardness, toughness, and cementing power. The gravel should show, on mechanical analysis, a grading of material which will contain sufficient stone of the larger size to insure stability and wearing qualities under traffic, and which will contain sufficient finer material to insure a proper bond. The best material will be composed of gravel graded so that it will have a maximum density.

Though the most available material will have a wide range as to sizes, the Committee believes that the following specification for sizes, adopted by the American Society of Municipal Improvements in 1916, may be followed safely:

"Two mixtures of gravel, sand, and clay shall be used, hereinafter designated in these specifications as No. 1 product (for top course) and No. 2 product (for middle and bottom courses).

"No. 1 product shall consist of a mixture of gravel, sand, and clay, with the proportions of the various sizes as follows: All to pass a 1½-in. screen and to have at least 60 and not more than 75 per cent. retained on a ¼-in. screen; at least 25 and not more than 75 per cent. of the total coarse aggregate (material over ¼ in. in size) to be retained on a ¾-in. screen; at least 65 and not more than 85 per cent. of the total fine aggregate (material under ¼ in. in size) to be retained on a 200-mesh sieve.

"No. 2 product shall consist of a mixture of gravel, sand, and clay, with the proportions of the various sizes as follows: All to pass a 2½-in. screen and to have at least 60 and not more than 75 per cent. retained on a ¼-in. screen; at least 25 and not more than 75 per cent. of the total coarse aggregate to be retained on a 1-in. screen; at least 65 and not more than 85 per cent. of the total fine aggregate to be retained on a 200-mesh sieve."

With gravel such a quartz, the cementation of which is low, a highly cementitious void filler is desirable, and a moderate quantity of clay or loam may be permissible.

A more generous use of gravel, especially in the surfacing of earth roads, should be encouraged. The low first cost and ease of maintenance should help materially to increase the mileage of serviceable roads in the country districts.

In drafting specifications, the refinements to be used in the methods of construction will depend on the kind and amount of traffic to be sustained, the character and quality of gravel to be secured in that particular locality, and other local conditions. Generally, however, the refinements should be carried to the point where further refinement would not be economical.

SHEET-ASPHALT PAVEMENTS[1]

General.—A sheet-asphalt wearing course, consisting of predetermined graded sand, filler, and asphalt cement, should be laid to a compacted thickness of not less than 1½ in. and not more than 2 in., on a binder course of bituminous concrete consisting of broken stone or broken stone and sand mixed with asphalt cement, the binder course having a compacted thickness of not less than 1 in. nor more than 1½ in.

Materials.—For heavy or medium traffic, the so-called close binder should be used, instead of the open binder, as the former possesses greater inherent stability than the latter. Specifications for the grading of open binder should be similar to those for the aggregate for Bituminous Concrete Pavements, Class *A;* and, for a close binder, similar to the following:

[1] For discussion of rates of grade, crown, artificial foundation, etc., see "General Conclusions," page 439.

Ninety-five per cent. of the binder aggregate shall pass a screen having circular openings the diameter of which shall be of three-quarters the thickness of the binder course to be laid. The remaining 5 per cent. shall not exceed in their smallest dimension the thickness of the binder course to be laid. The binder aggregate shall be graded from coarse to fine, so as to have the following mesh composition:

Passing 10-mesh sieve	15 to 35 per cent.	Total passing ½-in. screen, 35 to 85 per cent.
Passing ½-in. screen and retained on 10-mesh sieve	20 to 50 per cent.	

The sand for the wearing course shall be carefully graded. For pavements to be subjected to medium or heavy traffic, there should be a preponderance of the finer particles; and for pavements to be subjected to light traffic, there may be a preponderance of the coarser particles. Specifications for a sand for wearing courses to be subjected to medium or heavy traffic should be similar to the following: The sand shall be hard, clean, and moderately sharp. On sifting it shall have the following mesh composition:

Passing 200-mesh	0 to 5 per cent.	Total passing 80-mesh and retained on 200-mesh, 20 to 40 per cent.
Passing 100-mesh and retained on 200-mesh	10 to 25 per cent.	
Passing 80-mesh and retained on 100-mesh	6 to 20 per cent.	
Passing 50-mesh and retained on 80-mesh	5 to 40 per cent.	
Passing 40-mesh and retained on 50-mesh	5 to 30 per cent.	
Passing 30-mesh and retained on 40-mesh	5 to 25 per cent.	Total passing 10-mesh and retained on 40-mesh, 12 to 45 per cent.
Passing 20-mesh and retained on 30-mesh	5 to 15 per cent.	
Passing 10-mesh and retained on 20-mesh	2 to 15 per cent.	

The filler should be thoroughly dry limestone dust, or dust from other equally satisfactory stone, or Portland cement, the whole of which should pass a 30-mesh sieve and at least 66 per cent. of which should pass a 200-mesh sieve. The surface mixture should contain from 6 to 20 per cent. of this filler, depending on the kind of sand and asphalt used and the traffic conditions on the street or streets to be paved.

The specifications should contain detailed requirements covering the physical properties of the asphalt cement, and should prescribe the bitumen content of the binder course and sheet-asphalt wearing course mixture. For the grading mentioned, the bitumen should be, for the close binder, from 4 to 7 per cent.; and for the sheet-asphalt wearing course mixture, from 9.5 to 13.5 per cent.

Construction.—The broken stone for the binder should be heated to a temperature between 107°C. (225°F.) and 177°C. (350°F.). The sand when mixed with the asphalt cement should have a temperature between 135°C. (275°F.) and 190°C. (375°F.). The asphalt cement when used should have a temperature between 121°C. (250°F.) and 177°C. (350°F.).

The asphalt cement and broken stone, or broken stone and sand, for the binder course, and the asphalt cement, sand, and filler for the wearing course, should be thoroughly mixed by machinery until a uniform mixture is produced in which all the particles are thoroughly coated with asphalt cement.

When brought to the work, the temperature of the binder mixture should be between 93°C. (200°F.) and 163°C. (325°F.), and of the wearing course mixture between 110°C. (230°F.) and 177°C. (350°F.). The binder course and the wearing surface should be compacted separately by rolling with a self-propelled roller weighing not less than 200 lb. per inch of width of tread, the rolling being carried on continuously at the rate of not more than 200 sq. yd. per hour per roller until a satisfactory compression is obtained. Excessive use of water on the steam roller while compacting the courses of the pavement should not be permitted. During the rolling of the wearing course, a small quantity of Portland cement should be swept over its surface. In cases where sheet-asphalt is constructed next to the curb, it is advisable to coat the surface for a space of 12 in. next to the curb with hot asphalt cement.

STONE BLOCK PAVEMENTS[1]

Materials.—The stone pavements of this country are generally of granite or sandstone, the particular kind being determined by the availability of the different materials. Limestone is used to a certain extent in one or two cities, but so slightly that it need not be considered.

In order to make a good paving block, stone should be resistant to wear, hard and tough, and of such a character as to be easily broken into regular shapes. Toughness is more important than hardness, as in very few cases will the blocks of a stone pavement, of a character such as is generally used, be much reduced under actual traffic. The quantity of wear is not as important as that the wear shall be uniform, so that the surface of the pavement may be kept smooth and even.

The character of sandstone is such that, though it does wear smooth, it is never slippery; but with granite, if the stone is too hard, even when no particular wear is noticed under traffic, the surface soon becomes smooth and slippery, and the harder the stone, the more slippery it becomes.

To make suitable paving blocks, granite should be a medium and uniform-grained stone, of such a character that when broken it will present smooth and even surfaces, and have a percentage of wear of not more than 4.5 and toughness of not less than 8. It should have a crushing strength of not less than 20,000 lb. per sq. in.

Sandstone should be hard and tough, and of a character to meet the requirements given for granite, except as to crushing strength, which should be not less than 16,000 lb. per sq. in.

As the stone must be made into comparatively small blocks, and as this is done by expensive labor, the size of the blocks is extremely important. Probably the ideal sized block would be 8 in. long, 3½ in. wide, and, under general conditions, 5 in. deep, but if blocks were made to conform exactly to these dimensions, they would be extremely expensive, so that it is considered good practice to allow variations in length from 8 to 12 in., in width from 3½ to 4½ in., and in depth from 4¾ to 5½ in., and on light-traffic streets, where certain conditions make a stone pavement desirable, an even less depth may be permitted. When sandstone is used, the blocks may be a little wider, as far as use is concerned. All blocks, however, should be sorted, so that the adjacent courses can be kept as nearly uniform in width as possible. Under no circumstances, however, should blocks of different widths be used in the same course. The blocks should be dressed so that they will lie with close joints, and have good, smooth, and even heads.

Another form of stone block pavement, used to some extent in Europe, and which has recently been introduced into this country, is known as "Durax" in England and as "Kleinpflaster" in Germany. It consists of blocks approximating cubes 2½ to 4 in. in size, although they should not be exactly cubical; they should be sufficiently irregular, both in size and shape, to permit them to be laid in arcs of circles of comparatively small radii and so that the joints will not be excessively large. By laying the courses in circular arcs, none of the joints is parallel to any line of traffic.

In Europe these blocks are used to a great extent in resurfacing the broken stone roads where the traffic is too heavy for macadam, and to some extent in city streets. If the blocks can be produced in this country at a reasonable price, they will make very satisfactory roads. They have been used to a slight extent in pavements in some Southern cities.

During the last few years many pavements have been laid with granite blocks made by splitting up old ones which had been in use for some years. With blocks that ranged from 4 to 5 in. in width, 10 to 12 in., and even 14 in., in length, and 8 in. in depth, it has been found possible to get many good blocks of smaller size by cutting them up. The new blocks, being small, could be cut to a reasonably true surface without much work, with the result that the old blocks recut would actually lay more square yards in a pavement than the original ones. This practice is to be commended, both on the score of economy and result.

Foundation and Cushion.—It is assumed that the foundation for permanent stone pavements will in all cases be concrete. On the concrete must be spread a material to act both as a cushion to the blocks themselves and to even up the surface of the concrete; and the smoother the surface and the less the variation in the depth of the blocks, the thinner can be the cushion, although it should not be less than ¾ in., in any event.

The cushion which has generally been used for this purpose is sand, but recently engineers

[1] For discussion of rates of grade, crown, artificial foundation, joints, etc., see "General Conclusions," page 439.

have been considering the advisability of using Portland cement mortar instead. The objection to mortar, made by some, is that it makes a too solid base for the blocks, not giving any resiliency. This at present is a mooted question, and the Committee does not desire to express a positive opinion as to the relative values of the two. If a good bituminous cushion could be provided, it would probably be more satisfactory than either the mortar or the sand, but it is questionable if the advantage gained would justify the increase in expense. (See also Brick Pavements.)

Construction.—The blocks should be laid on the cushion stone to stone, keeping the joints as small as possible. The joints should be filled with water-proof material. For this purpose a Portland cement grout, asphalt, or some other bituminous filler is generally used, and in some cases sand is mixed with a bituminous material in order to increase the toughness of the filler. All these fillers give good results, but with cement grout the cost of taking up and restoring the pavement over cuts is increased over that of a bituminous filler, as in many cases blocks are broken in taking them up, and it is difficult to clean the cement from the individual blocks and also to keep the traffic from the cut or patch while the grout is setting after the pavement has been restored. Another disadvantage of the cement grout filler is that it is highly important that it be perfectly set before traffic is allowed on the pavement, and in large cities it is almost impossible to keep traffic from the pavement, after it is laid, for the necessary length of time.

WOOD BLOCK PAVEMENTS[1]

General.—In using wood in pavements, special attention should be given to the crown of the street, as this material undoubtedly presents, under certain conditions, a more slippery surface to traffic than any other. Wherever the longitudinal grade is sufficient to allow the water to run off freely, the crown should be very flat, not exceeding 3 in. in a roadway width of 30 ft. On streets which must be used continuously, the maximum grade allowed should not exceed 2 per cent., although on residence streets, where pavements can be avoided when they are exceptionally slippery, grades up to 3 or 4 per cent. are permissible. In the Middle West, where, as a rule, there is less moisture in the air under ordinary conditions, the foregoing grades have been exceeded with satisfactory results.

Kinds of Wood for Blocks.—Whatever the kind of wood used for pavements, it must be treated with some preservative, in order to make it suitable. It is important that as many kinds of wood be utilized as possible, so that in any territory the most available one can be used. Just how many varieties can be utilized is uncertain at the present time, but those that are undoubtedly good are: Southern yellow pine, Douglas fir, tamarack, Norway pine, hemlock, and black gum. In the East and Central West, Southern yellow pine, and on the Pacific Coast, Douglas fir, are generally used. Experiments will be necessary to determine just what other kinds will be satisfactory.

The blocks must be sound, and must be well manufactured, square-butted, square-edged, free from unsound, loose or hollow knots, knot holes, worm holes, and other defects, such as shakes, checks, etc., that would be detrimental to the blocks.

The number of annual rings in the 1 in. which begins 2 in. from the pith of the block should not be less than 6, measured radially, provided, however, that blocks containing between 5 and 6 rings in this inch may be accepted if they contain 33⅓ per cent., or more, of summer wood. In case the block does not contain the pith, the 1 in. to be used shall begin 1 in. away from the ring which is nearest to the heart of the block. The blocks in each charge shall contain an average of at least 70 per cent. of heart wood. No one block shall be accepted that contains less than 50 per cent. of heart wood.

Size of Blocks.—The blocks should be from 5 to 10 in. long, but should preferably average two times the depth. The Committee recommends blocks 4 in. in depth for very heavy traffic streets; blocks 3½ in. in depth for moderate traffic streets. For light traffic streets 3 in. in depth may be used, but where 3-in. blocks are used, no blocks should be longer than 8 in. They may be from 3 to 4 in. in width, but, in any one city block, all of them should be on uniform width. A variation of 1/16 in. should be allowed in the depth and ⅛ in. in width of the blocks from that specified. In all cases the width should be greater or less than the depth by at least ¼ in.

Preservatives for Wood Blocks.—Many different materials have been used in the past for wood preservatives, but the Committee believes that, taking all things into consideration,

[1] For discussion of rates of grade, crown, artificial foundation, etc., see "General Conclusions, page 439."

coal-tar creosote oil is the best. Some engineers, however, feel that a creosote oil produced from water-gas tar is as good as one produced from coal-gas tar, if not better. As the object of the preservative is not only to prevent the blocks from decay, but also to prevent them from swelling in wet weather or shrinking in dry weather, whatever the preservative, it should be of a character that will render the blocks stable and free from decay, for as long a time as possible. It is probable that under the traffic that prevails on most of the streets paved with wood in this country, if the blocks can be kept stable and free from decay, the pavement will last from 30 to 35 years, or even longer. It is necessary, however, to have an oil that is in itself stable and will remain in the blocks a long time.

It is thought that a heavy gravity oil will do this better than one of light gravity, as the former is less volatile and will maintain its condition better while exposed to atmospheric changes. The Committee recognizes that good results have been obtained by the use of a pure distillate oil, and also one which contains a certain quantity of coal-gas tar.

Treatment of Wood Blocks.—The timber may be either air-seasoned or green, but should preferably be treated within 3 months from the time it is sawed. Green timber and seasoned timber, however, should not be treated together in the same charge.

In any charge, blocks should contain at least 16 lb. of water-free oil per cubic foot of wood at the completion of the treatment. The blocks after treatment should show satisfactory penetration of the preservative, and in all cases the oil must be diffused throughout the sapwood. To determine this, at least 25 blocks shall be selected from various parts of each charge and sawed in half perpendicular to the fibers through the center, and if more than one of these blocks shows untreated sapwood, the charge should be re-treated. After re-treating, the charge should be again subjected to a similar inspection.

The surface of the blocks after treatment should be free from deposits of objectionable substances, and all blocks that have been materially warped, checked, or otherwise injured in the process of treatment, should be rejected.

Handling Blocks After Treatment.—Blocks should preferably be laid in the street as soon as possible after being treated. If they cannot be laid immediately, provision should be made to prevent them from drying out by stacking in close piles and covering them, and, if possible, by sprinkling them thoroughly at intervals. In any case, where they are not laid as soon as they are received on the street, they should be well sprinkled about 2 days before being laid, under the direction of the purchaser. It is important to have the wood sufficiently wet to be swelled to its maximum size before it is laid.

Inspection.—All material specified and processes used in the manufacture of the blocks therefrom should be subject to inspection, acceptance, or rejection at the plant of the manufacturer, which should be equipped with the necessary gauges, appliances, and facilities to enable the inspector to satisfy himself that the requirements of the specifications are fulfilled.

The purchaser should have the further right to inspect the blocks after delivery on the street, for the purpose of rejecting those that do not meet these specifications, except that the plant inspections should be final with respect to the kind of wood, rings per inch, oil, and treatment.

Construction.—There are two methods of laying wood blocks, one with and one without a cushion. In Europe it is invariably the practice to lay the blocks directly on the concrete bed. When this is done it is necessary that the surface of the concrete be made absolutely smooth and true to the required cross-section of the pavement. In this country the practice has been to surface-up the concrete, as has been mentioned in connection with stone blocks, with cement mortar or sand. The Committee believes that the cement mortar will give a better result than the sand. It should, however, be mixed as dry as possible and at the same time insure setting, and the blocks should be thoroughly rolled into it. If the sand cushion is used, the blocks should also be rolled to a smooth surface.

The Committee looks with a great deal of favor on the practice of finishing the concrete to a true surface and laying the blocks directly on it, and would suggest that engineers laying this pavement try this method, and, if the cost of producing a smooth concrete surface is not excessive, that the method be generally adopted.

The blocks should be laid closely and the joints filled with some suitable material. Three materials have been used in this country for joint filling: sand, cement grout, and a bituminous material. On heavy-traffic streets, if fine sand is used, good results will be obtained. On light-traffic streets, however, it may be better to use a bituminous filler of practically the same character as that used for granite. If a bituminous filler is used, the pavement should be covered with a thin layer of fine sand, which should be allowed to remain 1 or 2 weeks after the traffic has been allowed on the street. The Committee does not feel that

cement grout should be used, in any case. Though a wood block pavement should keep stable, it is undoubtedly safer to use a bituminous joint along the curb to provide for expansion or contraction.

Bleeding.—Considerable inconvenience has been caused in certain cities by "bleeding," or the exudation of the preservative on the surface of the street, after the blocks have been laid. If the proper precaution is taken with the character of the material and the character of the treatment, it is thought that this can be avoided. If the pavement, however, should bleed to such an extent as to be a nuisance, it should be covered with fine sand, so that the surface material can be absorbed. After one or two applications there should be no further trouble.

DEFINITIONS

For the sake of uniformity and clearness, the Committee recommends that the following lists of terms of frequent use in expressions relating to highway work be recognized as having the meanings set forth in the list, unless otherwise definitely stated at any time by a user of such term or terms.

In the list given will be found some terms and definitions adopted by the American Society for Testing Materials noted thus *, others by the American Reporters on Communication No. 10 at the Third International Road Congress, designated thus †, and other terms and definitions which have been proposed by the Committee on "Standard Tests for Road Materials" (Committee D-4) of the American Society for Testing Materials, which have been indicated thus ‡. The Committee wishes to acknowledge here its obligation for suggestions thus reaching it.

Aggregate.—The inert material, such as sand, gravel, shell, slag, or broken stone, or combinations thereof, with which the cementing material is mixed to form a mortar or concrete.

Asphalt.—†Solid or semi-solid native bitumens, solid or semi-solid bitumens obtained by refining petroleums, or solid or semi-solid bitumens which are combinations of the bitumens mentioned with petroleums or derivatives thereof, which melt on the application of heat, and which consist of a mixture of hydrocarbons and their derivatives of complex structure, largely cyclic and bridge compounds.

Asphalt Block Pavement.—One having a wearing course of previously prepared blocks of asphaltic concrete.

Asphalt Cement.—A fluxed or unfluxed asphaltic material, especially prepared as to quality and consistency, suitable for direct use in the manufacture of asphaltic pavements, and having a penetration of between 5 and 250.

Asphaltenes.—†The components of the bitumen in petroleum, petroleum products, malthas, asphalt cements, and solid native bitumens, which are soluble in carbon disulphide, but insoluble in paraffin naphthas.

Asphaltic.—Similar to, or essentially composed of, asphalt.

Base.—Artificial foundation.

Binder.—(1) A foreign or fine material introduced into the mineral portion of the wearing surface for the purpose of assisting the road metal to retain its integrity under stress, as well as, perhaps, to aid in its first construction. (2) The course, in a sheet-asphalt pavement, frequently used between the concrete foundation and the sheet-asphalt mixture of graded sand and asphalt cement.

Bitumen.—* A mixture of native or pyrogenous hydrocarbons and their non-metallic derivatives, which may be gases, liquids, viscous liquids, or solids, and which are soluble in carbon disulphide.[1]

Bituminous Cement.—A bituminous material suitable for use as a binder having cementing qualities which are dependent mainly on its bituminous character.

Bituminous Concrete Pavement.—One composed of broken stone, broken slag, gravel, or shell, with or without sand, Portland cement, fine inert material, or combinations thereof, and a bituminous cement incorporated together by a mixing method.

Bituminous Macadam Pavement.—One having a wearing course of macadam with the interstices filled by a penetration method with a bituminous binder.

Bituminous Material.—Material containing bitumen as an essential constituent.

Liquid Bituminous Material.—‡ Bituminous material showing a penetration at normal temperature under a load of 50 grams applied for 1 sec. of more than 350.

Semi-solid Bituminous Material.—‡ Bituminous material showing a penetration at normal temperature under a load of 100 grams applied for 5 sec. of more than 10, and under a load of 50 grams applied for 1 sec. of not more than 350.

Solid Bituminous Material.—‡ Bituminous material showing a penetration at normal temperature under a load of 100 grams applied for 5 sec. of not more than 10.

Bituminous Pavement.—One composed of broken stone, broken slag, gravel, shell, sand, or fine inert material, or combinations thereof, and bituminous cement incorporated together.

Bituminous Surface.—A superficial coat of bituminous material with or without the addition of stone or slag chips, gravel, sand, or material of similar character.

Blanket.—See "Carpet."

Bleeding.—The exudation of bituminous material on the roadway surface after construction.

Blown Petroleums.—† Semi-solid or solid products produced primarily by the action of air upon originally fluid native bitumens which are heated during the blowing process.

Bond.—The combined action of inertia, friction, and of the forces of adhesion and cohesion which helps the separate particles composing a crust or pavement to resist separation under stress. Mechanical bond is the bond produced almost wholly, in a well-built broken-stone macadam road, by the interlocking of angular fragments of stone and the subsequent filling of the remaining interstices with the finer particles.

Bound.—Bonded.

Water-bound.—Bonded with the aid of water.

Bituminous Bound.—Bonded with the aid of bituminous material.

Brick Pavement.—One having a wearing course of paving bricks or blocks.

Bridge.—A structure for the purpose of carrying traffic over a gap in the roadbed measuring 10 ft. or more in the clear span.

Camber of a Bridge.—The rise of its center above a straight line through its ends.

Camber of a Road.—See "Crown."

Carbenes.—† The components of the bitumen in petroleums, petroleum products, malthas, asphalt cements, and solid native bitumens, which are soluble in carbon disulphide but insoluble in carbon tetrachloride.

Carpet.—A bituminous surface of appreciable thickness, generally formed on top of a roadway by the application of one or more coats of bituminous material with gravel, sand, or stone chips added.

Cement.—An adhesive substance used for uniting particles of other materials to each other. Ordinarily applied only to calcined "cement rock," or to artificially prepared, calcined, and ground mixtures of limestone and silicious materials. Sometimes used to designate bituminous binder used in bituminous pavements, when the expression "bituminous cement" (q. v.) is understood to be meant.

Cement-Concrete.—An intimate mixture of gravel, shell, slag, or broken stone particles with certain proportions of sand or similar material, cement, and water, made previous to placing.

Cement-Concrete Pavement.—One having a wearing course of hydraulic cement concrete.

Cemented.—Bonded. Referring to water-bound macadam, the term "cemented" is used to designate that condition existing when, after rolling the stone forming the crust, the remaining voids have been filled with the finer sizes, and the stone dust or "flour" has, under the action of water, taken a "set," as does cement itself.

Chips.—Small angular fragments of stone or slag containing no dust.

Clay.—Finely divided earth, generally silicious and aluminous, which will pass a 200-mesh sieve. Also see "Gravel."

Coal-Tar.—†The mixture of hydrocarbon distillates, mostly unsaturated ring compounds, produced in the destructive distillation of coal.

Coat.—See "Carpet." (1) The total result of one or more single surface applications. (2) To apply a coat.

Coke-oven Tar.—† Coal-tar produced in by-product coke ovens in the manufacture of coke from bituminous coal.

Consistency.—* The degree of solidity or fluidity of bituminous materials.

Course.—One or more layers of road metal spread and compacted separately for the formation of the road or pavement. Courses are usually referred to in the order of their laying as first course, second course, third course, etc. Also a single row of blocks in a pavement.

Crown.—The rise in cross-section from the lowest to the highest part of the finished roadway. It may be expressed either as so many inches (or tenths of a foot), or as a rate per foot of distance from side to center, *i.e.*, "the crown is 4 in.," or "the crown is ½ in. to the foot."

Crusher Run.—The total unscreened product of a stone crusher.

Crusher-run Stone.—The product of a stone-crusher, unscreened except for the removal of the particles smaller than remaining on about a ¼-in. screen.

Crust.—That portion of a macadam or similar roadway above the foundation consisting of the road metal proper with its bonding agent or binder.

Culvert.—A structure for the purpose of carrying traffic over a gap in the road-bed, measuring less than 10 ft. in clear span.

Cut-back Products.—Petroleum, or tar residua, which have been fluxed, each with its own or similar distillates.

Dead Oils.—* Oils, with a density greater than water, which are distilled from tars.

Dehydrated Tars.—† Tars from which all water has been removed.

Ditch.—The open-side drain of a roadway, usually deep in proportion to its width, and unpaved.

Drainage.—Provision for the disposition of water.

Side-drainage.—That along the sides of the roadway.

Sub- or Under-drainage.—That below the surface.

Surface Drainage.—That on the roadway or ground surface.

V-drainage.—That provided by the construction of troughs in the sub-grade of the roadway which troughs are like a "V," with flat sloping sides, and are filled with stone.

Dust.—Earth or other matter in fine, dry particles, so attenuated that they can be raised and carried by air currents. The product of the crusher passing through a fine sieve.

Dust Layer.—Material applied to a roadway for temporarily preventing the formation or dispersion under traffic of distributable dust.

Earth Road.—A roadway composed of natural earthy material.

Emulsion.—A combination of water and oily material made miscible with water through the action of a saponifying or other agent.

Expansion Joint.—A separation of the mass of a structure, usually in the form of a joint filled with elastic material, which will provide opportunity for slight movement in the structure.

Fat.—Containing an excess. A far asphalt mixture is one in which the asphalt cement is in excess and the excess is clearly apparent.

Filler.—(1) Relatively fine material used to fill the voids in the aggregate. (2) Material used to fill the joints in a brick or block pavement.

Fixed Carbon.—* The organic matter of the residual coke obtained upon burning hydrocarbon products in a covered vessel in the absence of free oxygen.

Flour.—† Finely ground rocks or minerals pulverized to an impalpable product.

Flush Coat.—See "Seal Coat."

Flushing.—(1) Completely filling the voids. (2) Washing a pavement with an excess of water.

Flux.—† Bitumens, generally liquid, used in combination with harder bitumens for the purpose of softening the latter.

Footway.—The portion of the highway devoted especially to pedestrians. A sidewalk.

Foundation.—The portion of the roadway below and supporting the crust or pavement.

Artificial Foundation.—That layer of the foundation especially placed on the sub-grade for the purpose of reinforcing the supporting power of the latter itself, and composed of material different from that of the sub-grade proper.

Free Carbon.—* In tars, organic matter which is insoluble in carbon disulphide.

Gas-house Coal-tar.—† Coal-tar produced in gas-house retorts in the manufacture of illuminating gas from bituminous coal.

Grade.—(1) The profile of the center of the roadway, or its rate of rise or fall. (2) Elevation. (3) To establish a profile by cuts and fills or earthwork. (4) To arrange by sizes, broken stone, gravel, sand, or combinations of such materials.

Gravel.—Small stones or pebbles usually found in natural deposits more or less intermixed with sand, clay, etc., but in which mixture the particles which will not pass a 10-mesh sieve predominate.

Pea Gravel.—Clean gravel the particles of which approximate peas in size.

Grit.—Stone chips, slag chips, or small gravel.

Gutter.—The artificially surfaced and generally shallow waterway provided usually at the sides of the roadway for carrying surface drainage. Occasionally used synonymously with

"ditch," but incorrectly so, as "gutters" are always paved or otherwised surfaced, and ditches are not.

Haunches.—The sides or flanks of a roadway. Sometimes also called "quarters."

Highway.—The entire right of way devoted to public travel, including the sidewalks and other public spaces, if such exist.

Layer.—A course made in one application.

Loam.—Finely divided earthy material containing a considerable proportion of organic matter.

Macadam.—A road crust composed of stone or similar material broken into irregular angular fragments compacted together so as to be interlocked and mechanically bound to the utmost possible extent.

Mastic.—A mixture of bituminous material and fine mineral matter suitably made for use in highway construction and for application in a heated condition.

Mat.—† See "Carpet."

Matrix.—* The binding material or mixture of binding material and fine aggregate in which the large aggregate is embedded or held in place.

Mesh.—The square opening of a sieve.

Metal.—See "Road-metal."

Mortar.—A mixture of fine material such as sand, cement, and water or other liquid suitably proportioned and incorporated together for the purpose for which it is used.

Normal Temperature.—‡ As applied to laboratory observations of the physical characteristics of bituminous materials, is 25°C. (77°F.).

Oil-gas Tars.—* Tars produced by cracking oil vapors at high temperatures in the manufacture of oil gas.

Palliative.—A short-lived dust layer.

Patching.—Repairing or restoring small isolated areas in the surface of the metaled or paved portion of the highway.

Pavement.—The wearing course of the roadway or footway, when constructed with a cement or bituminous binder, or composed of blocks or slabs, together with any cushion or "binder" course.

Penetration.—In laboratory investigations, the distance, expressed in hundredths of a centimeter, entered a sample by a No. 2 cambric needle, operated in a machine for the purpose, and under known conditions of loading, time, and temperature. Where the conditions of test are not specifically mentioned, the load, time, and temperature are understood to be 100 grams, 5 sec., and 25°C. (77°F.), respectively, and the units of penetration to indicate hundredths of a centimeter.

Penetration Method.—The method of constructing a bituminous-macadam pavement by pouring or grouting the bituminous material into the upper course of the road metal before the binding of the latter has been completed.

Pitch.—† Solid residue produced in the evaporation or distillation of bitumens, the term being usually applied to residue obtained from tar.

Hard Pitch.—Pitch showing a penetration of not more than ten.

Soft Pitch.—Pitch showing a penetration of more than ten.

Straight-run Pitch.—‡ A pitch run in the initial process of distillation, to the consistency desired without subsequent fluxing.

Pocket.—A hole or depression in the wearing course.

Pot-hole.—A hole extending below the wearing course.

Profile.—A longitudinal section of a highway, generally taken along the center line.

Quarters.—The four sections of equal width which, side by side, make up the total width of a roadway.

Raveling.—The loosening of the metal composing the crust.

Refined Tar.—† A tar freed from water by evaporation or distillation which is continued until the residue is of desired consistency or a product produced by fluxing tar residuum with tar distillate.

Renewals.—Extensive repairs over practically the whole surface of the metaled or paved portion of the highway.

Repairs.—The restoration or mending of a considerable amount of the metaled or paved portion of the highway, but not usually of a majority of the surface area. More extensive than "Patching" but less so than "Renewals."

Resurfacing.—The renewal of the surface of the crust or pavement.

Road.—A highway outside of an urban district.

Road-bed.—The natural foundation of a roadway.

Road Metal.—Broken stone, gravel, slag, or similar material used in road and pavement construction and maintenance.

Roadway.—That portion of a highway particularly devoted to the use of vehicles.

Rock Asphalt.—Sandstone or limestone naturally impregnated with asphalt.

Rock Asphalt Pavement.—A wearing course composed of broken or pulverized rock asphalt with or without the addition of other bituminous materials.

Sand.—Finely divided rock detritus the particles of which will pass a 10-mesh and be retained on a 200-mesh screen. Also see "Gravel."

Sand-clay Road.—A roadway composed of an intimate mixture of sand and clay.

Scarify.—To loosen and disturb superficially.

Screen.—In laboratory work an apparatus, in which the apertures are circular, for separating sizes of material.

Screenings.—Broken rock of a size that will pass through a 1½- to ¾-in. screen, depending on the character of the stone.

Seal Coat.—A final superficial application of bituminous material during construction to a bituminous pavement.

Setting Up.—As applied to bituminous material, the relative quick change which takes place after its application to a roadway, indicated by its hardening after cooling and exposure to atmospheric and traffic conditions, as opposed to the slower changes later occurring gradually and almost imperceptibly.

Shaping.—Trimming up and preparing a sub-grade preparatory to applying the first course of the road metal or artificial foundation.

Sheet-asphalt Pavement.—One having a wearing course composed of asphalt cement and sand of predetermined grading, with or without the addition of fine material, incorporated together by mixing methods.

Sheet Pavement.—A pavement free from frequent joints such as would accompany small slabs or blocks, and which has an appreciable thickness (say, in excess of 1 in. on the average) for its wearing course.

Shoulders.—The portion of the highway between the edges of the road metal or pavement and the gutters, slopes, or watercourses.

Side Drain.—See "Drainage."

Sidewalk.—The portion of the highway reserved for pedestrians.

Sieve.—In laboratory work an apparatus, in which the apertures are square, for separating sizes of material.

Silt.—Naturally deposited fine earthy material, which will pass a 200-mesh sieve.

Slag.—‡ Fused or partly fused compounds of silica in combination with lime or other bases, resulting in secondary products from the reduction of metallic ores.

Spalls.—Fragments broken off by a blow, irregular in shape, and of sufficient size to be comparable to the original mass.

Squegee.—A tool with a rubber or leather edge for scraping or cleaning hard surfaces, or for spreading and distributing liquid material over and into the superficial interstices of roadways.

Squegee Coat.—An application by means of the squegee.

Stone Block Pavement.—One having a wearing course composed of stone blocks quite or nearly rectangular in shape.

Street.—A highway in an urban district.

Sub-grade.—The upper surface of the native foundation on which is placed the road metal or the artificial foundation, in case the latter is provided.

Superficial Coat.—A light surface coat.

Surface Coat.—See "Carpet."

Surface Treatment.—Treating the finished surface of a roadway with bituminous material.

Surfacing.—(1) The crust or pavement. (2) Constructing a crust or pavement. (3) Finally finishing the surface of a roadway. (4) Treating the surface of a finished roadway with a bituminous material.

Tailings.—Stones which after going through the crusher do not pass through the largest openings of the screens.

Tar.—† Bitumen which yields pitch upon fractional distillation and which is produced as a distillate by the destructive distillation of bitumens, pyro-bitumens, or organic material.

Telford.—Properly an artificial foundation advocated by Thomas Telford (1757–1820), and consisting of a pavement of stone about 8 in. thick, laid by hand, and closely packed and wedged together. The individual stones were desired to be about 16 sq. in. in section.

and about 8 in. in length. They were set close togehter on the prepared sub-grade, their longest dimension vertical and on their larger ends, their interstices chinked with smaller stones, and the whole rammed (or rolled) until firm and unyielding.

Telford Macadam.—Macadam with an artificial foundation of Telford.

Under-drain.—See "Drainage."

Up-keep.—Maintenance.

V-drain.—See "Drainage."

Viscosity.—‡ The measure of the resistance to flow of a bituminous material, usually stated as the time of flow of a given quantity of the material through a given orifice.

Volatile.—Applied to those fractions of bituminous materials which will evaporate at climatic temperatures.

Water-bound.—Bound or bonded with the aid of water.

Water-gas Tars.—* Tars produced by cracking oil vapors at high temperatures in the manufacture of carburetted water-gas.

Wearing Coat.—The superficial layer of the crust or pavement exposed to traffic.

Wearing Course.—The course of the crust or pavement exposed to traffic.

Wood Block Pavement.—One having a wearing course composed of wood paving blocks, generally rectangular in shape.

The Committee wishes to express again its deep appreciation of the assistance rendered it by the Board of Direction and by your Secretary, Dr. Chas. Warren Hunt, as well as by members of the Society and others.

Very respectfully,
For the Special Committee on Materials
for Road Construction and on
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FORM OF RECORD FOR DATA CONCERNING THE USE OF HIGHWAY MATERIALS

(The following forms of records are recommended for the use of highway engineers. They cover both bituminous and non-bituminous materials. Although combined in a single table, it is believed that the data for different kinds of road surfaces can advantageously be placed on separate sheets or cards for convenient filing and reference.)

GENERAL INFORMATION

State.....County.....Town or City.....
Road or street.....Limits of improvement.....
Length of improvement, in feet.....
Width of crust or pavement, in feet (average).....
Area of crust or pavement, in square yards.....
Kind of surface and foundation.....
Percentage of grade, maximum—per cent.....Minimum—per cent.....
Amount of crown, maximum....., minimum....., Nature of sub-grade.....
Maximum and minimum air temperature during year.....
Hours of working day.....Labor wage per hour.....
Contractor.....
Dates of beginning and completion of improvement.....
Class of highway or nature of traffic.....
TRAFFIC CENSUS FOR ..... HOURS, BEING THE AVERAGE OF .....
OBSERVATIONS TAKEN BETWEEN THE HOURS OF..... AND.....
ON.....
LOCATION OF POINT OF OBSERVATION.....

	Commercial vehicles			Passenger vehicles
	Empty	Loaded	Estimate (in pounds) of maximum load per inch of tire	
One-horse vehicles				
Two or three-horse vehicles				
Four or more horse vehicles				
Motor cycles				
Motor runabouts				
Motor touring cars (open or closed)				
Motor busses				
Motor trucks				

CONSTRUCTION AND COST DETAILS

A.—Foundation

1. Material
2. Thickness
3. Cost per square yard
4. Estimated life, in years

B.—Wearing Course

1. Material
2. Thickness
3. Size of block or brick
4. Kind and amount of bituminous cement
5. Kind of joint
6. Proportions of aggregate
7. Cushion or binder course
8. First cost per square yard
9. Life, in years
10. Average annual maintenance cost per square yard during life of wearing course

C.—Traffic Data

1. Tons per year (2000 lb.)
2. Average tons per yard of width
3. Proportion of tonnage on metal tires
4. Proportion of *C*-3 on tires 2 in. or less in width

ITEMS OF COST FOR EACH SQUARE YARD

	Foundation	Wearing course
Materials		
Labor		
Superintendence		
Overhead, including interest on plant, depreciation, etc.		

DATA PER SQUARE YARD

A-3.—First cost of foundation.....
A-5.—Annual interest and sinking fund for foundation.....
A-6.—Total annual cost of foundation.....
B-10.—Annual maintenance cost of wearing course.....
B-11.—Annual interest and sinking fund for wearing course.....
B-12.—Total annual cost of wearing course.....

Yearly cost per 1000 tons of traffic $= \frac{A\text{-}6 + B\text{-}12}{C\text{-}2} 1000 =$ .....

(The following data or such parts as apply to a particular road or street could be incorporated on the sheet or card containing the data immediately preceding, or could be placed on a separate sheet or card containing the results of tests and analyses.)

Broken Stone and Broken Slag

Name and origin	.....
Specific gravity	.....
Absorption of water per cubic foot	.....
Abrasion, percentage of loss	.....
Toughness	.....
Cementation	.....
Crushing strength per square inch	.....
Mechanical analysis	(Use table under this title)
Voids, percentage of, loose and compacted	.....

Gravel

Location	.....
Specific gravity	.....
Abrasion, percentage of loss	.....
Cementation	.....
Mechanical analysis	(Use table under this title)
Voids, percentage of, loose and compacted	.....

Sand

Location	.....
Specific gravity	.....
Mechanical analysis	(Use table under this title)
Voids, percentage of, loose and compacted	.....
Tensile strength in cement briquettes, as compared with standard Ottawa sand	.....

Mixtures of Sand or Other Fine Highway Materials with Broken Stone, Broken Slag, or Gravel

Specific gravity	.....
Mechanical analysis	(Use table under this title)
Voids, percentage of, loose and compacted	.....

Paving Brick

Composition	.....
Name of manufacturer	.....
Rattler test, percentage of loss	.....

Stone Block

Name and origin	.....
Specific gravity	.....
Absorption of water per cubic foot	.....
Abrasion, percentage of loss	.....
Toughness	.....
Hardness	.....
Crushing strength per square inch	.....

Wood Block

Character of wood	
Weight of blocks	
Soundness	
Rings per radial inch	
Quantity of preservative	
Absorption of water after treatment	
Character of preservative:	
Specific gravity at 25°C. (77°F.)	
Specific gravity at 38°C. (100°F.)	
Solubility in benzol or chloroform	
Water content	
Distillation:	
Up to 170°C	
170° to 200°C	
200° to 210°C	
210° to 235°C	
235° to 270°C	
270° to 300°C	
300° to 315°C	
315° to 355°C	

Mechanical Analysis	Percentages by weight		
Passing 200-mesh sieve			
Passing 100-mesh sieve, retained on 200-mesh sieve			
Passing 80-mesh sieve, retained on 100-mesh sieve			
Passing 50-mesh sieve, retained on 80-mesh sieve			
Passing 40-mesh sieve, retained on 50-mesh sieve			
Passing 30-mesh sieve, retained on 40-mesh sieve			
Passing 20-mesh sieve, retained on 30-mesh sieve			
Passing 10-mesh sieve, retained on 20-mesh sieve			
Passing ¼-in. screen, retained on 10-mesh sieve			
Passing ½-in. screen, retained on ¼-in. screen			
Passing ¾-in. screen, retained on ½-in. screen			
Passing 1 -in. screen, retained on ¾-in. screen			
Passing 1¼-in. screen, retained on 1 -in. screen			
Passing 1½-in. screen, retained on 1¼-in. screen			
Passing 2 -in. screen, retained on 1½-in. screen			
Passing 2½-in. screen, retained on 2 -in. screen			
Passing 3 -in. screen, retained on 2½-in. screen			
Passing 3½-in. screen, retained on 3 -in. screen			

Portland Cement

Loss on ignition, percentage	
Insoluble residue, percentage	
Specific gravity	
Retained on 200-mesh sieve, percentage	
Retained on 100-mesh sieve, percentage	
Steam test	
Initial set, time, in minutes	
Final set, time, in minutes	
Tensile strength, neat, 24 hours	
Tensile strength, neat, 7 days	
Tensile strength, 1-3 Ottawa sand, 7 days	
Tensile strength, 1:3 Ottawa sand, 28 days	
Compressive strength, per square inch	
Constancy of volume	

Bituminous Materials

The following forms are given as illustrations of those to be used for recording the properties of bituminous materials

Asphalt Cements for Bituminous Macadam, Bituminous Concrete, Asphalt Block and Sheet-asphalt Pavements and Fillers for Brick and Stone Block Pavements

Trade name
Manufacturer
General characteristics
Specific gravity at 25°C. (77°F.)
Flash point
Solubility in CS_2 (carbon disulphide)
  Organic matter insoluble
  Inorganic matter insoluble
Solubility of bitumen in CCl_4 (carbon tetrachloride)
Solubility of bitumen in petroleum naphtha
Penetration 4°C. (39°F.), 200 grams, 1 min.
Penetration 25°C. (77°F.), 100 grams, 5 sec.
Penetration 46°C. (115°F.), 50 grams, 5 sec.
Float test
Melting point by ring and ball method
Ductility at 4°C. (39°F.)
Ductility at 25°C. (77°F.)
Fixed carbon content
Paraffin content
Loss on evaporation at 163°C. (325°F.), 5 hours
  Penetration of residue, 4°C. (39°F.), 200 grams, 1 min.
  Penetration of residue 25°C. (77°F.), 100 grams, 5 sec.
  Penetration of residue 46°C. (115°F.), 50 grams, 5 sec.
  Melting point of residue, by ring and ball method
  Float test on residue
  Ductility of residue at 4°C. (39°F.)
  Ductility of residue at 25°C. (77°F.)

Tar Cements for Bituminous Macadam and Bituminous Concrete Pavements and Fillers for Brick and Stone Block Pavements

Trade name
Manufacturer
General characteristics
Water
Specific gravity at 25°C. (77°F.)
Flash point
Solubility in CS_2 (carbon disulphide)
Specific viscosity, Engler
Melting point, by cube method
Float test
Distillation by weight and by volume
  Up to 110°C.
  110° to 170°C.
  170° to 235°C.
  235° to 270°C.
  270° to 300°C.
  Specific gravity of total distillate at 25°C. (77°F.)
  Melting point of residue, by cube method
  Float test on residue

TESTS OF NON-BITUMINOUS MATERIALS

It is recommended that the following methods for performing tests of non-bituminous material be adopted as standards:

SPECIFIC GRAVITY OF COARSE AGGREGATES[1]

The apparent specific gravity shall be determined in the following manner:

The sample, weighing 1000 grams and composed of pieces approximately cubical or spherical in shape and retained on a screen having 1.27-cm. (½-in.) circular openings, shall be dried to constant weight at a temperature between 100 and 110°C. (212 and 230°F.), cooled, and weighed to the nearest 0.5 gram. Record this weight as weight *A*. In the case of homogeneous material, the smallest particles in the sample may be retained on a screen having 1¼-in. circular openings.

Immerse the sample in water for 24 hours, surface-dry individual pieces with aid of a towel or blotting paper, and weigh. Record this weight as weight *B*.

Place the sample in a wire basket of approximately ¼-in. mesh, and about 12.7 cm. (5 in.) square and 10.3 cm. (4 in.) deep, suspend in water[2] from center of scale pan, and weigh. Record the difference between this weight and the weight of the empty basket suspended in water as weight *C*. (Weight of saturated sample immersed in water.)

The apparent specific gravity shall be calculated by dividing the weight of the dry sample (*A*) by the difference between the weights of the saturated sample in air (*B*) and in water (*C*), as follows:

$$\text{Apparent specific gravity} = \frac{A}{B - C}.$$

Attention is called to the distinction between apparent specific gravity and true specific gravity. Apparent specific gravity includes the voids in the specimen, and is, therefore, always less than or equal to, but never greater than, the true specific gravity of the material.

APPARENT SPECIFIC GRAVITY OF SAND, STONE SCREENINGS, OR OTHER FINE HIGHWAY MATERIAL

Apparatus.—The determination shall be made with a Jackson specific gravity apparatus which shall consist of a burette, with graduations reading to 0.01 in specific gravity, about 23 cm. (9 in.) long and with an inside diameter of about 0.6 cm. (0.25 in.), which shall be connected with a glass bulb approximately 13 cm. (5.5 in.) long and 4.5 cm. (1.75 in.) in diameter, the glass bulb being of such size that from a mark on the neck at the top to a mark on the burette just below the bulb, the capacity is exactly 180 c.c. (6.09 oz.); and an Erlenmeyer flask which shall contain a hollow ground-glass stopper having the neck of the same bore as the burette and a capacity of exactly 200 c.c. (6.76 oz.) up to the graduation on the neck of the stopper.

Method of Determination.—The method shall consist of: First, dry at not more than 110°C. (230°F.) to a constant weight a sample weighing about 55 grams; second, weigh to 0.1 gram, 50 grams of the dry sample and pour it into the unstoppered Erlenmeyer flask; third, fill the bulb and burette with kerosene, leaving just space enough to take the temperature by introducing a thermometer through the neck; fourth, remove the thermometer and add sufficient kerosene to fill exactly to the mark on the neck, drawing off any excess with the burette; fifth, run into the flask about one-half of the kerosene in the bulb to remove air bubbles and then run in more kerosene, removing any material adhering to the neck of the flask, until the kerosene is just below the ground glass; sixth, place the hollow ground-glass stopper in position and turn it to fit tightly, and then run in kerosene exactly to the 200-c.c. (6.76-oz.) graduation on the neck, care being taken to remove all air bubbles in the flask; seventh, read the specific gravity from the graduation on the burette, and the temperature of the oil in the flask, noting the difference between the temperature of the oil in the bulb before the determination and that of the oil in the flask after the determination; eighth, make a temperature correction to the reading of the specific gravity in accordance with the table furnished by the manufacturer of the apparatus, adding the correction if the temperature of the kerosene has increased and subtracting it if the temperature of the kerosene has decreased.

[1] Proposed in 1917 by Committee D-4, "Standard Tests for Road Materials," of the Am. Soc. for Testing Materials.

[2] The basket may be conveniently suspended by a fine wire hung from a hook shaped in the form of a question mark with the top end resting on the center of the scale pan.

ABSORPTION OF WATER PER CUBIC FOOT OF ROCK[1]

"The absorption of water per cubic foot of rock shall be determined by the following method: First, a sample weighing between 29 and 31 grams and approximately cubical in shape shall be dried in a closed oven for 1 hour at a temperature of 110°C. (230°F.) and then cooled in a desiccator for 1 hour; second, the sample shall be rapidly weighed in air; third, trial weighings in air and in water of another sample of approximately the same size shall be made in order to determine the approximate loss in weight on immersion; fourth, after the balances shall have been set at the calculated weight, the first sample shall be weighed as quickly as possible in distilled water having a temperature of 25°C. (77°F.); fifth, allow the sample to remain 48 hours in distilled water maintained as nearly as practicable at 25°C. (77°F.), at the termination of which time bring the water to exactly this temperature and weigh the sample while immersed in it; sixth, the number of pounds of water absorbed per cubic foot of the sample shall be calculated by the following formula:

$$\text{Pounds of water absorbed per cubic foot} = \frac{W_2 - W_1}{W - W_1} \times 62.24,$$

in which W = the weight in grams of sample in air, W_1 = the weight in grams of sample in water just after immersion, W_2 = the weight in grams of sample in water after 48 hours immersion, and 62.24 = the weight in pounds of a cubic foot of distilled water having a temperature of 25°C. (77°F.).

"Finally, the absorption of water per cubic foot of the rock, in pounds, shall be the average of three determinations made on three different samples according to the method above described."

ABRASION TEST FOR BROKEN STONE OR BROKEN SLAG[2]

"The machine shall consist of one or more hollow iron cylinders; closed at one end and furnished with a tightly fitting iron cover at the other; the cylinders to be 20 cm. (7.87 in.) in diameter and 34 cm. (13.38 in.) in depth inside. These cylinders are to be mounted on a shaft at an angle of 30° with the axis of rotation of the shaft.

"At least (13.6 kg.) 30 lb. of coarsely broken stone shall be available for a test. The rock to be tested shall be broken in pieces as nearly uniform in size as possible, and as nearly 50 pieces as possible shall constitute a test sample. The total weight of rock in a test shall be within 10 grams of 5 kilograms (11.02 lb.). All test pieces shall be washed and thoroughly dried before weighing. 10,000 revolutions, at the rate of between 30 and 33 to the minute, must constitute a test. Only the percentage of material worn off which will pass through a 0.16 cm. (1-16 in.) mesh sieve shall be considered in determining the amount of wear. * * *"

ABRASION TEST FOR GRAVEL

NOTE.—As tests to determine the loss on abrasion of gravel are in an experimental stage, the Committee has included two methods which have been used and are being investigated. In noting the results obtained, the Committee advises stating the method used.

Method No. 1.—The test for abrasion of gravel shall be made with a Deval abrasion machine. (See "Abrasion Test for Broken Stone or Broken Slag.")

A charge of gravel shall consist of pieces which shall pass a screen having circular openings 5.08 cm. (2 in.) in diameter and be retained on a screen having circular openings 1.27 cm. (½ in.) in diameter. The total weight of gravel in a charge shall be within 10 grams of 5 kg. (11.02 lb.). The gravel to compose a charge shall be washed, and dried in a closed oven for 1 hour at a temperature within 5° of 110°C. (230°F.). The charge of gravel shall be placed in one cylinder of the machine, which shall be rotated at a rate of not less than 30 nor more than 33 rev. per min. Ten thousand revolutions shall constitute a test. The percentage of material worn off which will pass through a sieve having openings of 0.16 cm. (1/16 in.) shall be considered the amount of wear of the charge of gravel. The loss by abrasion, determined as stated, shall be expressed in terms of the percentage of the total weight of the charge of gravel.

[1] Proposed in 1914 by Committee D-4, "Standard Tests for Road Materials," of the Am. Soc. for Testing Materials.

[2] Method adopted by the Am. Soc. for Testing Materials, August 15, 1908.

Method No. 2.[1]—The aggregate is first screened through screens having circular openings 2 in., 1 in., and ½ in. in diameter. The sizes used for this test are divided equally between those passing the 2-in. and retained on the 1-in. screen, and those passing the 1-in. and retained on the ½-in. screen. The material of these sizes is washed and dried. The following weights of the dried stone are then taken: 2500 grams of the size passing the 2-in. and retained on the 1-in. screen, and 2500 grams of the size passing the1-in. and retained on the ½-in. screen. This material is placed in the cast-iron cylinder of the Deval machine, as specified for the standard abrasion on stone. Briefly described, this machine consists of a frame and two or more cylinders mounted at an angle of 30° with the axis of rotation. The cylinders are 20 cm. in diameter and 34 cm. deep, inside dimensions. Six cast-iron spheres, 1.875 in. in diameter and weighing approximately 0.95 lb. (0.45 kg.) each, are placed in the cylinder as an abrasive charge. (The iron composing these spheres is the same as that used for the spheres in the Standard Paving Brick Rattler Test.) After the cast-iron spheres have been placed in the cylinder the lid is bolted on and the cylinder is mounted in the frame of the Deval machine. The duration of the test and the rate of rotation are the same as specified for the standard test for stone, namely, 10,000 revolutions at a rate of from 30 to 33 rev. per min. At the completion of the test the material is taken out and screened through a 16-mesh sieve. The material retained on the sieve is washed and dried and the percentage of loss by abrasion of the material passing the 16-mesh sieve is calculated. When the material has a specific gravity of less than 2.20, a total weight of 4000 grams, instead of 5000 grams, shall be used in the abrasion test.

TOUGHNESS TEST FOR ROCK OR SLAG[2]

Definition.—Toughness, as applied to rock, is the resistance offered to fracture under impact, expressed as the final height of blow required of a standard hammer to cause fracture of a cylindrical test specimen of given dimensions.

Sampling.—Quarry samples of rock from which test specimens are to be prepared shall measure at least 6 in. on a side and at least 4 in. in thickness, and, when possible, shall have the plane of structural weakness[3] of the rock plainly marked thereon. Samples should be taken from freshly quarried material, and only from pieces which show no evidences of incipient fracture due to blasting or other causes. The samples should preferably be split from large pieces by the use of plugs and feathers, and not by sledging. Commercial stone-block samples from which test specimens are to be prepared shall measure at least 3 in. on each edge.

Size and Form of Test Specimen.—Specimens for test shall be cylinders prepared as described in the next paragraph, 25 mm. in height and from 24 to 25 mm. in diameter. Three test specimens shall constitute a test set. The ends of the specimens shall be plane surfaces at right angles to the axis of the cylinder.

Preparation of Test Specimens.—One set of specimens shall be drilled perpendicular and another parallel to the plane of structural weakness of the rock, if such plane is apparent. If a plane of structural weakness is not apparent, one set of specimens shall be drilled at random. Specimens shall be drilled in a manner which will not subject the material to undue stresses and will insure the specified dimensions.[4] The ends of the cylinders may be sawed with a band or diamond saw,[5] or in any other way which will not induce incipient fracture, but shall not be chipped or broken off with a hammer. After sawing, the ends of the specimens shall be ground plane with carborundum or emery on a cast-iron lap until the cylinders are 24 mm. in length.

Impact Machine.—Any form of impact machine which will comply with the following essentials may be used in making the test: A cast-iron anvil weighing not less than 50 kg.,

[1] Proposed by the First Conference of State Highway Testing Engineers and Chemists held at the U. S. Office of Public Roads in February, 1917.

[2] Proposed in 1917 by Committee D-4, "Standard Tests for Road Materials," of the Am. Soc. for Testing Materials.

[3] The plane of structural weakness, in certain cases, may be the rift, cleavage, or bedding plane.

[4] The form of diamond drill described in *Bulletin* No. 347, U. S. Department of Agriculture, pp. 6–7, is recommended, and should prove satisfactory, if the instructions are strictly followed.

[5] A satisfactory form of diamond saw is described in *Bulletin* No. 347, U. S. Department of Agriculture, pp. 7–9.

firmly fixed on a solid foundation; a hammer weighing 2 kg., arranged so as to fall freely between suitable guides; a plunger made of hardened steel, and weighing 1 kg., arranged to slide freely in a vertical direction in a sleeve, the lower end of the plunger being spherical in shape, with a radius of 1 cm.; means for raising the hammer and for dropping it on the plunger from any specified height from 1 to not less than 75 cm., and means for determining the height of fall to approximately 1 mm.; means for holding the cylindrical test specimen securely on the anvil without rigid lateral support, and under the plunger in such a way that the center of its upper surface, throughout the test, shall be tangent to the spherical end of the plunger at its lowest point.

Method of Testing.—The test shall consist of a 1-cm. fall of the hammer for the first blow, a 2-cm. fall for the second blow, and an increase of 1-cm. fall for each succeeding blow, until failure of the test specimen occurs.

Recording and Reporting Results.—The height of the blow, in centimeters, at failure shall be the toughness of the test specimen. The individual and the average toughness of three test specimens shall be reported when no plane of structural weakness is apparent. In cases where a plane of structural weakness is apparent, the individual and average toughness of the three specimens in each set shall be reported and identified. Any peculiar condition of a test specimen which might affect the result, such as the presence of seams, fissures, etc., shall be noted and recorded with the test result.

HARDNESS TEST FOR ROCK OR SLAG

The test for hardness shall be made with a "Dorry," or similar machine, consisting of a revolving disk on which is fed, at a uniform rate, a standard quartz sand passing a 30- and retained on a 40-mesh sieve. Two cores, each 25 mm. (0.98 in.) in diameter, shall be cut from the material to be tested, and their faces ground off so as to be at right angles to the long axes of the cores. The cores shall be placed in the holders or dies and weighted so that the entire weight of each core with its holder and added weight is 1250 grams. Each core shall be ground in the machine on one face for 1000 revolutions, after which it shall be reversed and ground on the other face for an equal number of revolutions. The loss of weight of each specimen shall be determined at the end of each 1000 revolutions, and the average loss in weight shall be used for stating the hardness of the material, which latter shall be expressed by the formula: Hardness $= 20 - \frac{1}{3}W$, where W equals the average loss, in grams per 1000 revolutions.

CEMENTATION OF ROCK, SLAG, AND GRAVEL POWDERS

The cementation test shall be made as follows: Of the material to be tested, 500 grams shall be broken to pass a 1.27-cm. ($\frac{1}{2}$-in.) mesh sieve and then placed in a ball mill with 90 c.c. (3.04 oz.) of water and two steel shot weighing together 9 kg. (20 lb.). The mill and its charge shall be revolved for $2\frac{1}{2}$ hours at a rate of 2000 rev. per hour. The dough thus formed shall then be removed, and 25 grams of an average sample of it shall be placed in a metal die, 25 mm. (0.98 in.) in diameter, and subjected to a pressure of 132 kg. per sq. cm. for an instant in a hydraulic press. The cylindrical briquette resulting should measure exactly 25 mm. (0.98 in.) in height. If it does not, subsequent samples of the dough shall be taken in such quantity that the resulting briquette after compression will be exactly 25 mm. (0.98 in.) in height. Five such briquettes shall be made and allowed to dry in the air for a period of 20 hours, after which they shall be heated for 4 hours in a hot-air oven at a temperature of 93.3°C. (200°F.), and then cooled in a desiccator for 20 min. These cylinders or briquettes shall then be tested in a machine, as follows:

The machine shall be arranged so that a 1-kg. (2.20-lb.) hammer is raised to a height of 1 cm. (0.39 in.) and then falls freely on a plunger transmitting the shock of the blows of the hammer through the plunger to the test piece, successive blows being struck by the hammer at a rate of 40 to 70 per min., until the test piece fails, which is indicated by the failure of the plunger or hammer to rebound. The test piece shall be placed on the anvil under the plunger without lateral support, and may be fastened in place on the anvil by a drop of shellac. The average of the number of blows on the five briquettes, required to produce failure in each case, is the result to be reported, and is the "coefficient of cementation."

CRUSHING STRENGTH OF ROCK OR SLAG

Cylinders shall be cut from a suitable block of the material to be tested, each of which cylinders shall be, as nearly as practicable, 5 cm. (2 in.) in diameter and 10 cm. (4 in.) in

length. After cutting, the dimensions of each cylinder shall be accurately measured and recorded. Each cylinder shall then be subjected to compression, and the ultimate stress at which its failure occurs shall be noted. This stress divided by the average area in cross-section of the cylinder in square inches shall be reported. It is desirable that the test of the material shall be made on at least three such cylinders separately, and the average of the three or more specimens shall be taken as the average resistance to crushing of the material. In making the test, the cylinder shall be fixed in the testing machine so as to be unsupported on its sides and rest squarely on its ends, and the compressive stress shall be applied cumulatively. The ends of the cylinder shall be at right angles to its long axis, and the blocks or pieces of the machine in contact with the ends of the cylinder and through which the pressure is transmitted shall have such position and freedom of movement in the machine as will insure the application of the stress directly along or parallel to the long axis of the cylinder.

MECHANICAL ANALYSIS OF BROKEN STONE, BROKEN SLAG, OR GRAVEL[1]

The method shall consist of, first, drying at not more than 110°C. (230°F.), to a constant weight, a sample weighing in pounds six times the diameter in inches of the largest holes required; second, passing the sample through such of the following sized screens having circular openings as are required or called for by the specification, the screens to be used in the order named: 8.89 cm. (3½ in.), 7.62 cm. (3 in.), 6.35 cm. (2½ in.), 5.08 cm. (2 in.), 3.81 cm. (1½ in.), 3.18 cm. (1¼ in.), 2.54 cm. (1 in.), 1.90 cm. (¾ in.), 1.27 cm. (½ in.), and 0.64 cm. (¼ in.); third, determining the percentage by weight retained on each screen; fourth, recording the mechanical analysis in the following manner:

Percentage passing 0.64-cm. (¼-in.) screen..... =
Percentage passing 1.27-cm. (½-in.) screen and retained on 0.64-cm. (¼-in.) screen..... =
Percentage passing 1.90-cm. (¾-in.) screen and retained on 1.27-cm. (½-in.) screen..... =
Percentage passing 2.54-cm. (1-in.) screen and retained on 1.90-cm. (¾-in.) screen..... =
..... =

100.00

MECHANICAL ANALYSIS OF SAND OR OTHER FINE HIGHWAY MATERIAL

The method shall consist of: First, drying at not more than 110°C. (230°F.) to a constant weight a sample weighing 50 grams; second, passing the sample through each of the following mesh sieves, the sieves to be used in the order named:

Meshes per linear inch (2.54 cm.)	Diameter of wire	
	Inches	Millimeters
10	0.027	0.6858
20	0.0165	0.4191
30	0.01375	0.34925
40	0.01025	0.26035
50	0.009	0.22865
80	0.00575	0.1460
100	0.0045	0.1143
200	0.00235	0.05969

third, determining the percentage by weight retained on each sieve, the sifting being continued on each sieve until less than 1 per cent. of the weight retained on each sieve shall pass

[1] Method adopted in 1916 by the Am. Soc. for Testing Materials.

through the sieve during the last minute of sifting; fourth, recording the mechanical analysis in the following manner:

Percentage passing 200-mesh sieve	=
Percentage passing 100-mesh sieve and retained on 200-mesh sieve	=
Percentage passing 80-mesh sieve and retained on 100-mesh sieve	=
Percentage passing 50-mesh sieve and retained on 80-mesh sieve	=
	=
	100.00

MECHANICAL ANALYSIS OF MIXTURES OF SAND OR OTHER FINE HIGHWAY MATERIAL WITH BROKEN STONE, BROKEN SLAG, OR GRAVEL

The method shall consist of: First, drying at not more than 110°C. (230°F.) to a constant weight, a sample weighing in pounds six times the diameter in inches of the largest holes required; second, separating the sample by the use of a 10-mesh sieve (American Society for Testing Materials standard sieve); third, examining the portion retained on the 10-mesh sieve in accordance with the method for making a "Mechanical Analysis of Broken Stone, Broken Slag, or Gravel;" fourth, examining the portion passing the 10-mesh sieve in accordance with the method for making a "Mechanical Analysis of Sand or Other Fine Highway Material;" fifth, recording the mechanical analysis in the following manner:

Percentage passing 200-mesh sieve	=
Percentage passing 100-mesh sieve and retained on 200-mesh sieve	=
Percentage passing 80-mesh sieve and retained on 100-mesh sieve	=
	=
Percentage passing 10-mesh sieve and retained on 20-mesh sieve	=
Percentage passing 0.64-cm. (¼-in.) screen and retained on 10-mesh sieve	=
Percentage passing 1.27-cm. (½-in.) screen and retained on 0.64-cm. (¼-in.) screen	=
Percentage passing 1.90-cm. (¾-in.) screen and retained on 1.27-cm. (½-in.) screen	=
	=
	= 100.00

VOIDS IN MINERAL AGGREGATES[1]

"The voids in mineral aggregates shall be determined by the Cone Specific Gravity Method. In the method of making the determination of voids, as hereinafter described, there shall be used a truncated cone made of No. 18, B. & S.-gage galvanized steel with caulked seams, and having the following dimensions: over-all diameter of bottom, 25.4 cm. (10 in.); over-all height, 25.4 cm. (10 in.); inside diameter of opening, 7.6 cm. (3 in.). The test shall be made in the following manner: First, thoroughly mix the aggregate by rolling on paper; second, fill the cone with aggregates, avoiding segregation; third, compact aggregate in cone by oscillation on edge of cone resting on wooden floor, wooden box, or block of wood, and use cotton waste pressed against surface of aggregate to prevent segregation during oscillation; fourth, continue to add aggregate and compact until the cone is full of thoroughly compacted aggregate, which process will require from 300 to 500 oscillations; fifth, weigh cone with aggregate; sixth, weigh cone empty; seventh, weigh cone full of clean water; eighth, determine the specific gravity of aggregate; ninth, the percentage of voids in the aggregate shall be calculated by the following formula:

$$\text{Percentage of voids} = \left(1 - \frac{C - A}{(B - A)\,D}\right)100$$

in which A = the weight in grams of the cone; B = the weight in grams of the cone filled with water; C = the weight in grams of the cone filled with compacted aggregate; D = the specific gravity of the aggregate."

[1] Proposed in 1915 by Committee D-4, "Standard Tests for Road Materials," of the Am. Soc. for Testing Materials.

RATTLER TEST FOR PAVING BRICK

Construction of the Rattler

General Design.—The machine shall be of good mechanical construction, self-contained, shall conform to the following details of material and dimensions, and shall consist of barrel, frame, and driving mechanisms as herein described.

The Barrel.—The barrel of the machine shall be made up of the head, head-liners, staves, and stave-liners.

The Frame and Driving Mechanism.—The barrel shall be mounted on a cast-iron frame of sufficient strength and rigidity to support it without undue vibration. It shall rest on a rigid foundation with or without the interposition of wooden plates, and shall be fastened thereto by bolts at not less than four points. It shall be driven by gearing having a ratio of driver to driven of not less than one to four.

The Abrasive Charge.—The abrasive charge shall consist of cast-iron spheres of two sizes. When new, the larger spheres shall be 9.52 cm. (3.75 in.) in diameter and shall weigh approximately 3.40 kg. (7.5 lb.) each. Ten spheres of this size shall be used. These shall be weighed separately after each ten tests, and if the weight of any large sphere falls to 3.175 kg. (7 lb.), it shall be discarded and a new one substituted; provided, however, that all the large spheres shall not be discarded and substituted by new ones at any single time, and that, so far as possible, the large spheres shall compose a graduated series in various stages of wear. When new, the smaller spheres shall be 4.762 cm. (1.875 in.) in diameter and shall weigh approximately 0.43 kg. (0.95 lb.) each. In general, the number of small spheres in a charge shall not fall below 245 nor exceed 260. The collective weight of the large and small spheres shall be as nearly 136 kg. (300 lb.) as possible. No small sphere shall be retained in use after it has been worn down so that it will pass a circular hole 4.45 cm. (1.75 in.) in diameter, drilled in an iron plate 0.64 cm. (¼ in.) in thickness,[1] or weigh less than 0.34 kg. (0.75 lb.). Further, the small spheres shall be tested, by passing them over the above plate or by weighing, after ten tests, and any which pass through or fall below the specified weight, shall be replaced by new spheres; provided, further, that all the small spheres shall not be rejected and replaced by new ones at any one time, and that, so far as possible, the small spheres shall compose a graduated series in various stages of wear. At any time that any sphere is found to be broken or defective, it shall at once be replaced.

The iron composing these spheres shall have a chemical composition within the following limits:

Combined carbon	Not less than 2.50 per cent.
Graphitic carbon	Not more than 0.25 per cent.
Silicon	Not more than 1.00 per cent.
Manganese	Not more than 0.50 per cent.
Phosphorus	Not more than 0.25 per cent.
Sulphur	Not more than 0.08 per cent.

Operation of the Test

The Brick Charge.—The number of bricks per test shall be ten for all bricks of so-called "block-size," having dimensions which fall between 20.32 and 22.86 cm. (8 and 9 in.) in length, 7.62 and 9.52 cm. (3 and 3¾ in.) in breadth, and 9.52 and 10.8 cm. (3¾ and 4¼ in.) in thickness. No brick should be selected as part of a regular test that would be rejected by any other requirements of the specifications under which the purchase is made. (*Note by Committee.*—Each brick should be marked by small holes drilled in one of the faces of the brick, and the initial weight of each brick composing the charge should be determined.)

Speed and Duration of Revolution.—The rattler shall be rotated at a uniform rate of not less than 29.5 nor more than 30.5 rev. per min., and 1800 revolutions shall constitute the test. A counting machine shall be attached to the rattler for recording the revolutions. A margin of not more than 10 revolutions will be allowed for stopping. Only one start and stop per test is generally acceptable. If, from accidental causes, the rattler is stopped and started more than once during a test, and the loss exceeds the maximum permissible under the specifications, the test shall be discarded and another made.

[1] Adapted from the "Standard Specifications for Paving Brick," adopted in 1915 by the Am. Soc. for Testing Materials.

The Scales.—The scales shall have a capacity of not less than 136 kg. (300 lb.), shall be sensitive to 14.17 grams (0.5 oz.), and shall be tested by a standard test weight at intervals of not less than every ten tests.

The Results.—The loss shall be calculated in percentage of the initial weight of the brick composing the charge. In weighing the rattled brick, any piece weighing less than 0.45 kg. (1 lb.) shall be rejected. (*Note by Committee.*—The loss for each brick should also be calculated in percentage of the initial weight of each brick composing the charge.)

ABSORPTION OF WATER BY WOOD BLOCKS AFTER TREATMENT

Five blocks of average character shall be heated in an oven to a temperature of 110°C. (230°F.) for 3 hours, then weighed, and immersed in water for the same length of time. At the end of this time, they shall be taken out, wiped dry, and weighed, the difference in weight before and after immersion, calculated on the weight after heating, being the percentage of absorption.

CEMENT

For sampling, analysis, and testing of cement, the methods described in the "Final Report of the Special Committee on Uniform Tests of Cement" (*Trans. Am. Soc. Civ. Eng.*, **75** (1912), 665–696).

TESTS OF BITUMINOUS MATERIALS

It is recommended that the following methods for performing tests of bituminous materials be adopted as standards:

SPECIFIC GRAVITY

For liquid and semi-solid materials, some standard form of pycnometer shall be used. For solid materials, the suspension method shall be used. Material and distilled water shall have a temperature of 25°C. (77°F.).

The pycnometer to be used shall consist of a fairly heavy, straight-walled glass tube, 70 mm. (2.75 in.) long and 22 mm. (0.875 in.) in diameter, ground to receive a solid glass stopper with a hole of 1.6-mm. (0.063-in.) bore, in place of the usual capillary opening. The lower part of this stopper shall be made concave in order to allow all air bubbles to escape through the bore. The depth of the cup-shaped depression is 4.8 mm. (0.188 in.) at the center. The stoppered tube shall have a capacity of about 24 cu. cm. (0.811 oz.) and when empty shall weigh about 28 grams. Its principal advantages are: (1) that any desired quantity of bituminous material may be poured in without touching the sides above the level desired; (2) it is easily cleaned; (3) on account of the 1.6-mm. (0.063-in.) bore, the stopper can be more easily inserted when the tube is filled with a very viscous oil than if it contained a capillary opening. When testing solid or semi-solid materials with the pycnometer, extreme care should be taken in melting, to avoid loss by evaporation, and, in filling the pyknometer, to avoid entrapping air.

When working with semi-solid bituminous materials which are too soft to be broken and handled in fragments, the following method of determining their specific gravity has been used with good results. The clean, dry pycnometer is first weighed empty and this weight is called a. It is then filled in the usual manner with freshly distilled water at 25°C. (77°F.), and the weight is again taken and called b. A small quantity of the material is then placed in a spoon and brought to a fluid condition by the gentle application of heat, with care that no loss by evaporation occurs. When sufficiently fluid, enough is poured into the dry pyknometer, which may also be warmed, to fill it about half full, without allowing the material to touch the sides of the tube above the desired level. The tube and contents are then allowed to cool to room temperature, after which the tube, with the stopper, is carefully weighed. This weight is called c. Distilled water, at 25°C. (77°F.), is then poured in until the pyknometer is full. After this the stopper is inserted and the whole is cooled to 25°C. (77°F.) by a 30-min. immersion in a beaker of distilled water maintained at this temperature. All surplus moisture is then removed with a soft cloth, and the pycnometer and contents are weighed. This weight is called d. From the weights obtained, the specific gravity of the material may be readily calculated by the following formula:

$$\text{Specific gravity } \frac{25^\circ \text{ C. (77° F.)}}{25^\circ \text{ C. (77° F.)}} = \frac{c-a}{(b-a)-(d-c)}.$$

Both a and b are constants, and need be determined only once. It is necessary, therefore, to make only two weighings for each determination after the first. Results obtained according to this method are accurate to within two units in the third decimal place, whereas the open-tube method commonly used is accurate to the second decimal place only.

The specific gravity of fluid bituminous material may be determined in the ordinary manner with this pycnometer by completely filling it with the material and dividing the weight of the bituminous material thus obtained by that of the same volume of water.

FLASH POINT

The flash point shall be determined by the closed-cup test.

Although, for ordinary purposes, the open-cup method of determining the flash and burning points of bituminous materials is reasonably accurate, the closed-cup method described below is to be preferred.

The oil tester shall consist of a copper oil cup having a capacity of about 300 cu. cm. (10.1 oz.), and shall be heated in a water or oil bath by a small bunsen flame. The cup shall be provided with a glass cover carrying a thermometer, and a hole for inserting the testing flame. The testing flame shall be obtained from a jet of gas passed through the piece of glass tubing, and shall be about 5 mm. (0.197 in.) in length.

The flash test shall be made as follows: The oil cup shall first be removed and the bath filled with water or cottonseed oil. The oil may always be used, and is necessary for bituminous material flashing at a temperature of more than 100°C. (212°F.). The oil cup shall be replaced and filled with the material to be tested to within 3 mm. (0.118 in.) of the flange joining the cup and the vapor chamber above. The glass cover shall then be placed on the oil cup and the thermometer adjusted so that its bulk shall be just covered by the bituminous material. The bunsen flame shall be applied in such a manner that the temperature of the material in the cup shall be raised at the rate of about 5°C. (9°F.) per min. From time to time the testing flame shall be inserted in the opening in the cover to about half way between the surface of the material and the cover. The appearance of a faint bluish flame over the entire surface of the bituminous material will show that the flash point has been reached and the temperature at this point is taken.

SOLUBILITY IN CARBON DISULPHIDE (CS_2)

This test shall consist in dissolving the bituminous material in carbon disulphide and recovering any insoluble matter by filtering the solution through an asbestos felt. The Gooch crucible used for the determination shall be 4.4 cm. (1.722 in.) wide at the top, tapering to 3.6 cm. (1.417 in.) at the bottom, and shall be 2.5 cm. (0.984 in.) deep.

The asbestos shall be cut with scissors into pieces not exceeding 1 cm. (0.394 in.) in length, after which it shall be shaken up with just sufficient water to pour easily. The crucible shall be filled with the suspended asbestos and allowed to settle for a few moments. A light suction shall then be applied to draw off all the water and leave a firm mat of asbestos in the crucible. More of the suspended material shall be added, and the operation shall be repeated until the felt shall be so dense that it scarcely transmits light when held so that the bottom of the crucible is between the eye and the source of light. The felt shall then be washed several times with water, and drawn firmly against the bottom of the crucible by an increased suction. The crucible shall be removed to a drying oven for a few minutes, after which it shall be ignited at red heat over a bunsen burner, cooled in a desiccator, and weighed.

Two grams of bituminous material or 10 grams of an asphalt topping or rock asphalt shall then be placed in an Erlenmeyer flask, which shall have been weighed previously, and the accurate weight of the sample obtained. One hundred cubic centimeters (3.381 oz.) of chemically pure carbon disulphide shall be poured into the flask, in small portions, with continual agitation, until all lumps disappear and nothing adheres to the bottom. The flask shall then be corked and set aside for 15 min. to allow settlement of the insoluble material.

The weighed Gooch crucible containing the felt shall be set up over the dry pressure flask, and the solution of bituminous material in carbon disulphide shall be decanted through the felt without suction by gradually tilting the flask, with care not to stir up any precipitate that may have settled out. At the first sign of any sediment coming out, the decantation shall be stopped and the filter allowed to drain. A small quantity of carbon disulphide

shall then be washed down the sides of the flask, after which the precipitate shall be brought upon the felt and the flask scrubbed, if necessary, with a feather or "policeman," to remove all adhering material. The contents of the crucible shall be washed with carbon disulphide until the washings run colorless. Suction shall then be applied until there is practically no odor of carbon disulphide in the crucible, after which the outside of the crucible shall be cleaned with a small quantity of the solvent. The crucible and contents shall be dried in the hot-air oven at 100°C. (212°F.) for about 20 min., cooled in a desiccator and weighed. If any appreciable quantity of insoluble matter adheres to the flask, it shall also be dried and weighed, and any increase over the original weight of the flask shall be added to the weight of insoluble matter in the crucible. The total weight of insoluble material may include both organic and mineral matter. The former, if present, shall be burned off by ignition at a red heat until no incandescent particles remain, thus leaving the mineral matter or ash, which can be weighed on cooling. The difference between the total weight of material insoluble in carbon disulphide and the weight of substance taken equals the total bitumen, and the percentage weights are calculated and reported as total bitumen, and insoluble organic and inorganic matter, on the basis of the weight of material taken for analysis.

This method is quite satisfactory for straight oil and tar products, but, where native asphalts are present, it will be found practically impossible to retain all the finely divided mineral matter on an asbestos felt. It is generally more accurate, therefore, to obtain the result for total mineral matter by direct ignition of a 1-gramme sample in a platinum crucible, or to use the result for ash obtained in the fixed carbon test. The total bitumen is then determined by deducting from 100 per cent. the sum of the percentages of total mineral matter and insoluble organic matter. If the presence of a carbonate mineral is suspected, the percentage of mineral matter may be most accurately obtained by treating the ash from the fixed carbon determination with a few drops of ammonium carbonate solution, drying at 100°C. (212°F.), then heating for a few minutes at a dull red heat, cooling, and weighing again.

When difficulty in filtering is experienced—for instance, when Trinidad asphalt is present in any quantity—a longer period of subsidence than 15 min. is necessary, and the following method, adopted in 1911 by the American Society for Testing Materials is recommended:

Analysis of Sample.—After drying, from 2 to 15 grams (as may be necessary to insure the presence of 1 to 2 grams of pure bitumen) are weighed into a 150-c.c. tared Erlenmeyer flask, and treated with 100 c.c. of carbon disulphide. The flask is then loosely corked and shaken from time to time until all large particles of the material have been broken up. It is then set aside for 48 hours to settle. The solution is decanted into a similar flask that has been previously weighed. As much of the solvent is poured off as possible without disturbing the residue. The contents of the first flask are again treated with fresh carbon disulphide, shaken as before, and then put away with the second flask for 48 hours to settle.

The liquid in the second flask is then carefully decanted on a weighed Gooch crucible, 3.2 cm. in diameter at the bottom, fitted with an asbestos filter, and the contents of the first flask are similarly treated. The asbestos filter is made of ignited long-fiber amphibole, packed in the bottom of a Gooch crucible to the depth of not more than $\frac{1}{8}$ in. In filtering, no vacuum is to be used and the temperature is to be kept between 20° and 25°C. After passing the liquid contents of both flasks through the filter, the residue on the filter is thoroughly washed, and the residues remaining in them are shaken with more fresh carbon disulphide and allowed to settle for 24 hours, or until it is seen that a good subsidation has taken place. The solvent in both flasks is then again decanted through the filter, and the residues remaining in them are washed until the washings are practically colorless. All washings are to be passed through the Gooch crucible.

The crucible and both flasks are then dried at 125°C. and weighed. The filtrate containing the bitumen is evaporated, the bituminous residue burned, and the weight of the ash thus obtained added to that of the residue in the two flasks and the crucible. The sum of these weights deducted from the weight of substance taken gives the weight of soluble bitumen.

SOLUBILITY OF BITUMEN IN CARBON TETRACHLORIDE (CCl_4)

The test shall be conducted in exactly the same manner as described for the test for "Solubility in Carbon Disulphide," except that 100 c.c. (3.381 oz.) of chemically pure carbon tetrachloride shall be used in place of carbon disulphide, and the percentage of bitumen insoluble in carbon tetrachloride shall be reported on the basis of the bitumen

taken as 100, the quantity of bitumen having been determined by the method described under the heading "Solubility in Carbon Disulphide."

CONSISTENCY

The "Engler Viscosimeter," the "New York Testing Laboratory Float," or the "Penetrometer," shall be used, as practicable, at 4°C. (39°F.), 25°C. (77°F.), and 46°C. (115°F.).

VISCOSITY TEST

The viscosity of liquid bituminous materials shall be determined at any desired temperature by using the "Engler Viscosimeter." This apparatus consists of a brass vessel for holding the material to be tested, and is closed by a cover. To the conical bottom is fitted a conical outflow tube exactly 20 mm. (0.787 in.) long, with a diameter of 2.9 mm. (0.114 in.) on top, and of 2.8 mm. (0.110 in.) on the bottom. This tube is closed and opened by a pointed hardwood stopper. Pointed metal projections are placed on the inside of the vessel at equal distances from the bottom, and serve for measuring the charge of material, which is 240 cu. cm. (8.116 oz.). A thermometer is used to ascertain the temperature of the material to be tested. The vessel is surrounded by a brass jacket, which holds the material which may be used as a heating bath, either water or cottonseed oil, according to the temperature at which the test is to be made. A tripod serves as a support for the apparatus, and also carries a ring burner by which the bath is heated directly. The measuring cylinder, having a capacity of 100 cu. cm. (3.381 oz.), which is sufficiently accurate for work with road materials, is placed directly under the outflow tube.

As all viscosity determinations should be compared with that of water at 25°C. (77°F.), the apparatus shall have been previously calibrated as follows: The cup and outlet tube shall first be scrupulously cleaned. A piece of soft tissue paper is convenient for cleaning the tube. The stopper shall then be inserted in the tube, and the cup shall be filled with water at 25°C. (77°F.) to the top of the projections. The measuring cylinder shall be placed directly under the outflow tube so that the material, on flowing out, will not touch the sides. The stopper shall then be removed and the time required, both for 50 and 100 cu. cm. (1.691 and 3.381 oz.) to run out, shall be ascertained by using a stop-watch. The results thus obtained shall be checked a number of times. The time required for 50 cu. cm. (1.691 oz.) of water should be about 11 sec., and for 100 cu. cm. (3.381 oz.) about 22.8 sec.

Bituminous materials shall be tested in the same manner as water, and the temperature at which the test is made shall be controlled by the bath. The material shall be brought to the desired temperature and maintained there for at least 3 min. before making the test. The results are expressed as specific viscosity compared with water at 25°C. (77°F.), as follows:

$$\text{Specific viscosity at ——°C. for —— c.c.} = \frac{\text{second for passage of given volume at ——°C.}}{\text{seconds for passage of same volume of water at 25°C. (77° Fahr.)}}$$

FLOAT TEST

The float apparatus consists of two parts, an aluminum float or saucer and a conical brass collar. The two parts are made separately, so that one float may be used with a number of brass collars.

In making the test, the brass collar shall be placed with the small end down on the brass plate, which shall have been previously amalgamated with mercury by rubbing it first with a dilute solution of mercuric chloride or nitrate and then with mercury. A small quantity of the material to be tested shall be heated in the metal spoon until quite fluid, with care that it shall suffer no appreciable loss by volatilization and that it shall be kept free from air bubbles. It shall then be poured into the collar in a thin stream until slightly more than level with the top. After the material has cooled to room temperature, the surplus may be removed with a spatula blade which has been slightly heated. The collar and plate shall then be placed in one of the tin cups containing ice water maintained at 5°C. (41°F.), and left in this bath for 15 min. Meanwhile, the other cup shall be filled about three-fourths full of water and placed on the tripod, and the water shall be heated to any temperature desired for the test. This temperature shall be accurately maintained, and shall at no time throughout the entire test be allowed to vary more than 0.5°C. (0.9°F.)

from the temperature selected. After the material to be tested has been kept in the ice water for 15 min., the collar and contents shall be removed from the plate and screwed into the aluminum float, which shall then be immediately floated in the warmed bath. As the plug of bituminous material becomes warm and fluid, it is gradually forced upward and out of the collar, until water gains entrance to the saucer and causes it to sink.

The time, in seconds, between placing the apparatus on the water and when the float sinks shall be taken as a measure of the consistency of the material under examination.

PENETRATION TEST[1]

Apparatus.—The container for holding the material to be tested shall be a flat-bottomed, cylindrical dish, 55 mm. (2 3/16 in.) in diameter and 35 mm. (1 3/8 in.) deep.[2] The needle for this test shall be a cylindrical steel rod 50.8 mm. (2 in.) long and having a diameter of 1.016 mm. (0.04 in.) and turned on one end to a sharp point having a 6.35-mm. (1/4-in.) taper. The water bath shall be maintained at a temperature not varying more than 0.1°C. (0.18°F.) from 25°C. (77°F.). The volume of water shall be not less than 10 liters, and the sample shall be immersed to a depth of not less than 10 cm. (4 in.) and shall be supported on a perforated shelf not less than 5 cm. (2 in.) from the bottom of the bath. Any apparatus which will allow the needle to penetrate without appreciable friction, and which is accurately calibrated to yield results in accordance with the definition of penetration, will be acceptable. The transfer dish for the container shall be a small dish or tray of such capacity as will insure complete immersion of the container during the test. It shall be provided with some means which will insure a firm bearing and prevent rocking of the container.

Preparation of Sample.—The sample shall be completely melted at the lowest possible temperature, and stirred thoroughly until it is homogeneous and free from air bubbles. It shall then be poured into the sample container to a depth of not less than 15 mm. (5/8 in.). The sample shall be protected from dust and allowed to cool in an atmosphere not lower than 18°C. (65°F.) for 1 hour. It shall then be placed in the water bath along with the transfer dish and allowed to remain 1 hour.

Testing.—In making the test, the sample shall be placed in the transfer dish filled with water from the water bath of sufficient depth to cover the container completely. The transfer dish containing the sample shall then be placed on the stand of the penetration machine. The needle, loaded with specified weight (See Report Form for Asphalt Cement, page 468), shall be adjusted to make contact with the surface of the sample. This may be accomplished by making contact of the actual needle point with its image reflected by the surface of the sample from a properly placed source of light. Either the reading of the dial shall then be noted or the needle brought to zero. The needle is then released for the specified period of time, after which the penetration machine is adjusted to measure the distance penetrated. At least three tests shall be made at points on the surface of the sample not less than 1 cm. (3/8 in.) from the side of the container and not less than 1 cm. (3/8 in.) apart. After each test the sample and transfer dish shall be returned to the water bath, and the needle shall be carefully wiped toward its point with a clean, dry cloth, to remove all adhering bitumen. The reported penetration shall be the average of at least three tests the values of which shall not differ more than four points between maximum and minimum. When desirable to vary the temperature, time, and weight (See Report Form for Asphalt Cement, page 468), and, in order to provide for a uniform method of reporting results when variations are made, the samples shall be melted and cooled in air as above directed. They shall then be immersed in water or brine, as the case may require, for 1 hour at the temperature desired.

MELTING-POINT

Cube Method for Tar Cements

The material under examination shall be first melted in a spoon by the gentle application of heat until sufficiently fluid to pour readily. Care shall be taken that it suffers no appreciable loss by volatilization. It shall then be poured into a 12.7 mm. (0.5-in.) brass cubical mould, which shall have been amalgamated with mercury, and shall be placed on an amalgamated brass plate. The brass may be amalgamated by washing it first with a dilute solution

[1] Adopted in 1916 by the Am. Soc. for Testing Materials.
[2] Adopted in 1916 by the Am. Soc. for Testing Materials.

of mercuric chloride or nitrate, after which the mercury is rubbed into the surface. By this means the bituminous material is, to a considerable extent, prevented from sticking to the sides of the mould. The hot material shall slightly more than fill the mould, and, when cooled, the excess shall be cut off with a hot spatula.

After cooling to room temperature, the cube shall be removed from the mould and fastened on the lower arm of a No. 10 wire (B. & S. gage), bent at right angles at one end and suspended beside a thermometer in a covered Jena glass beaker having a capacity of 400 cu. cm. (13.526 oz.), which shall be placed in a water bath, or, for high temperatures, a cotton-seed-oil bath. The wire shall be passed through the center of two opposite faces of the cube, which shall then be suspended with its base 25.4 mm. (1 in.) above the bottom of the beaker. The water or oil bath shall consist of an 800-cu. cm. (27.051-oz.) low-form Jena glass beaker, suitably mounted for the application of heat from below. The beaker in which the cube is suspended shall be of the tall-form Jena type, without lip. The metal cover shall have two openings. A cork, through which passes the long arm of the wire, shall be inserted in one hole and the thermometer in the other. The bulb of the thermometer shall be just level with the cube and at an equal distance from the side of the beaker. In order that a reading of the thermometer may be made, if necessary, at the point which passes through the cover, the hole shall be triangular and covered with an ordinary object glass through which the stem of the thermometer may be seen. Readings made through this glass shall be calibrated to the angle of observation, which may be made constant by sighting always from the front edge of the opening to any given point on the stem of the thermometer below the cover.

After the test specimens shall have been placed in the apparatus, the liquid in the outer vessel shall be heated in such a manner that the thermometer registers an increase of 5°C. (9°F.) per min. The temperature at which the bituminous material touches a piece of paper placed in the bottom of the beaker shall be taken as the melting point. Determinations made in the manner described shall not vary more than 2°C. (3.6°F.) for successive trials on the same material. At the beginning of this test the temperature of both bituminous material and bath shall be approximately at 25°C. (77°F.).

RING AND BALL METHOD FOR ASPHALT CEMENTS[1]

The apparatus shall consist of a brass ring, 15.875 mm. (⅝ in.) in diameter, 6.35 mm. (¼ in.) deep, 2.38125 mm. (3/32 in.) wide, suspended 25.40 mm. (1 in.) above bottom of a beaker; a steel ball, 9.525 mm. (⅜ in.) in diameter, weighing between 3.45 and 3.50 grams; a standardized thermometer; a glass beaker, approximately 600-c.c. capacity.

Carefully melt the sample and fill the ring with material to be tested. Remove any excess. Place ball in center of ring and suspend in beaker containing approximately 400 c.c. of water at a temperature of 5°C. (41°F.). Arrange thermometer bulb within ½ in. of sample and at same level. Apply heat uniformly over bottom of beaker in quantity sufficient to raise temperature 5°C. (9°F.) per min. Record temperature at starting test and every minute thereafter until test is completed. The rate of heating is very important. Softening point is temperature at which specimen has dropped 1 in. Successive tests should average within 3°C. For temperatures above 95°C., glycerin shall be used instead of water.

LOSS ON EVAPORATION[2]

The amount lost by oils and asphaltic compounds when they are heated in an oven at a temperature of 163°C. (325°F.) plus or minus 1°C. (2°F.) shall be determined by heating 50 grams of the water-free substance contained in a flat-bottomed dish, the inside dimensions of which are approximately 2 3/16 in. in diameter and 1⅜ in. deep (3-oz. Gill style ointment box, deep style) for 5 hours. The oven in which the substance is heated shall be brought to the prescribed temperature before the sample is introduced, and the temperature of the sample under test shall be regarded as that of a similar quantity of the same material immediately adjoining it in the oven, in which the bulb of a standardized thermometer is immersed. The oven may be either of circular or rectangular form, and the source of heat either gas or electricity. The samples under test shall rest in the same relative position in a single row on a perforated circular shelf, 9¾ in. in diameter, suspended by a vertical

[1] Proposed in 1916 by Committee D-4, "Standard Tests for Road Materials," of the Am. Soc. for Testing Materials.

[2] Adopted in 1916 by the Am. Soc. for Testing Materials.

shaft midway in the oven, which is revolved by mechanical means at the rate of from 5 to 6 rev. per min. (Note.—If additional periods of heating are desired, it is recommended that they be made in successive increments of 5 hours each.) If the residue after heating is to be tested for penetration, the sample should be thoroughly mixed by stirring until it is cool, and thereafter manipulated in accordance with the directions of the standard test for penetration of bituminous materials.

DISTILLATION[1]

Note.—Equivalents in English units have been added by the Special Committee on Materials for Road Construction.

Sampling.—The sample as received shall be thoroughly stirred and agitated, warming, if necessary, to insure a complete mixture before the portion for analysis is removed.

Dehydration.—If the presence of water is suspected, or known, the material shall be dehydrated before distillation. About 500 cu. cm. (16.907 oz.) of the material is placed in an 800-cu. cm. (27.051-oz.) copper still provided with a distilling head connected with a water-cooled condenser. A ring burner is used, starting with a small flame at the top of the still, and gradually lowering it, if necessary, until all the water has been driven off. The distillate is collected in a 200-cu. cm. separatory funnel with the tube cut off close to the stop-cock. When all the water has been driven over and the distillate has settled out, the water is drawn off and the oils are returned to the residue in the still. The contents of the still shall have cooled to below 100°C. (212°F.) before the oils are returned, and they shall be well stirred and mixed with the residue.

Apparatus.—The apparatus shall consist of the following standard parts:

(*a*) *Flask.*—The distillation flask shall be a 250-cu. cm. Engler distilling flask, having the following dimensions:

Diameter of bulb	8.0 cm. (3.150 in.)
Length of neck	15.0 cm. (5.906 in.)
Diameter of neck	1.7 cm. (0.670 in.)
Surface of material to lower side of tubulature	11.0 cm. (4.331 in.)
Length of tubulature	15.0 cm. (5.906 in.)
Diameter of tubulature	0.9 cm. (0.354 in.)
Angle of tubulature	75°

A variation of 3 per cent. from the foregoing measurements will be allowed.

(*b*) *Thermometer.*—The thermometer shall conform to the following requirements:

It shall be made of thermometric glass of a quality equivalent to suitable grades of Jena or Corning make. It shall be thoroughly annealed. It shall be filled above the mercury with inert gas which will not act chemically on or contaminate the mercury. The pressure of the gas shall be sufficient to prevent separation of the mercury column at all temperatures of the scale. There shall be a reservoir above the final graduation large enough so that the pressure will not become excessive at the highest temperature. The thermometer shall be finished at the top with a small glass ring or button suitable for attaching a tag. Each thermometer shall have for identification the maker's name, a serial number, and the letters "A. S. T. M. Distillation."

The thermometer shall be graduated from 0 to 400°C. at intervals of 1°C. Every fifth graduation shall be longer than the intermediate ones, and every tenth graduation beginning at zero shall be numbered. The graduation marks and numbers shall be clear-cut and distinct.

The thermometer shall conform to the following dimensions:

Total length, maximum	385 mm.
Diameter of stem	7 mm.; permissible variation, 0.5 mm.
Diameter of bulb, minimum	5 mm.; and shall not exceed diameter of stem.
Length of bulb	12.5 mm.; permissible variation, 2.5 mm.
Distance from 0° to bottom of bulb	30 mm.; permissible variation, 5 mm.
Distance from 0° to 400°	295 mm.; permissible variation, 10 mm.

[1] Adopted in 1916 by the Am. Soc. for Testing Materials.

The accuracy of the thermometer when delivered to the purchaser shall be such that when tested at full immersion the maximum error from 0 to 200°C. shall not exceed the following:

From 0 to 200°C.	0.5°C.
From 200 to 300°C.	1.0°C.
From 300 to 375°C.	1.5°C.

The sensitiveness of the thermometer shall be such that when cooled to a temperature of 74°C. below the boiling point of water at the barometric pressure, at the time of test, and plunged into free flow of steam, the meniscus shall pass the point 10°C. below the boiling point of water in not more than 6 sec.

The thermometer shall be set up as for the distillation test, using water, naphthalene, and benzophenone as distilling liquids. The correctness of the thermometer shall be checked at 0 and 100°C. after each third distillation until seasoned.

(*c*) *Condenser.*—The condenser tube shall have the following dimensions:

Length	500 mm. (19.685 in.)
Width	12 to 15 mm. (0.472 to 0.591 in.)
Width of adaptor end	20 to 25 mm. (0.787 to 0.984 in.)

(*d*) *Stands.*—Two iron stands shall be provided, one with a universal clamp for holding the condenser, and one with a light grip arm with a cork-lined clamp for holding the flask.

(*e*) *Burner and Shield.*—A bunsen burner shall be provided, with a tin shield, 20 cm. (7.784 in.) long and 9 cm. (3.543 in.) in diameter. The shield shall have a small hole through which to observe the flame.

(*f*) *Cylinders.*—The cylinders used in collecting the distillate shall have a capacity of 25 cu. cm. (0.845 oz.), and shall be graduated in tenths of a cubic centimeter.

Setting up the Apparatus.—The apparatus shall be set up, the thermometers being placed so that the top of the bulb is opposite the middle of the tubulature. All connections shall be tight.

Method.—One hundred cubic centimeters (3.381 oz.) of the dehydrated material to be tested shall be placed in a tared flask and weighed. After adjusting the thermometer, shield, condenser, etc., the distillation is commenced, the rate being regulated so that 1 cu. cm. (0.034 oz.) passes over every minute. The receiver is changed as the mercury column just passes the fractionating point.

Up to 110°C. (230°F.)
110°C. to 170°C. (338°F.)
170°C. to 235°C. (455°F.)
235°C. to 270°C. (518°F.)
270°C. to 300°C. (572°F.)

To determine the quantity of residue, the flask is weighed again when distillation is complete. During the distillation the condenser tube shall be warmed when necessary, in order to prevent the deposition of any sublimate. The percentages of fraction should be reported, both by weight and by volume.

DUCTILITY

A briquette of the material to be tested shall be formed by pouring the molten material into a briquette mould. The dimensions of the briquette shall be: 1 cm. (0.394 in.) in thickness throughout its entire length; distance between the clips or end pieces, 3 cm. (1.181 in.); width of asphalt cement section at mouth of clips, 2 cm. (0.787 in.); width at minimum cross-section, half way between clips, 1 cm. (0.394 in.). The center pieces are removable, the briquette mould being held together during moulding with a clamp or wire.

The moulding of the briquette shall be done as follows; The two center sections shall be well amalgamated to prevent the asphalt cement from adhering to them, and the briquette mould shall then be placed on a freshly amalgamated brass plate. The asphalt cement to be tested, while in a molten state, shall be poured into the mould, a slight excess being added to allow for shrinkage on cooling. When the asphalt cement in the mould is nearly cool,

the briquette shall be cut off level, with a warm knife or spatula. When it is thoroughly cooled to the temperature at which it is desired to make the test, the clamp and the two side pieces are removed, leaving the briquette of asphalt cement held at each end by the ends of the mould, which now play the part of clips. The briquette shall be kept in water for 30 min. at 4°C. (39°F.) or 25°C. (77°F.) before testing, dependent on the temperature at which the ductility is desired. The briquette with the clips attached shall then be placed in a "ductility test machine" filled with water at one of the above temperatures to a sufficient height to cover the briquette not less than 50 mm.(1.969 in.). This machine consists of a rectangular water-tight box, having a movable block working on a worm-gear from left to right. The left clip is held rigid by placing its ring over a short metal peg provided for this purpose; the right clip is placed over a similar rigid peg on the movable block. The movable block is provided with a pointer which moves along a centimeter scale. Before starting the test, the centimeter scale is adjusted to the pointer at zero. Power is then applied by the worm-gear pulling from left to right at the uniform rate of 5 cm. (1.969 in.) per min. The distance, in centimeters, registered by the pointer on the scale at the time of rupture of the thread of asphalt cement shall be taken as the ductility of the asphalt cement.

SOLUBILITY IN PETROLEUM NAPHTHA.

Two grams of the material shall be placed in a 4-oz. oil-sample bottle, made up to 100 cu. cm. (3.381 oz.) with 88° Bé. petroleum naphtha (boiling point between 40°C. (104°F.) and 55°C. (131°F.)), and the whole well shaken until the sample is digested. The bottle shall then be centrifugalized for 10 min., 50 c.c. (1.691 oz.) withdrawn into a weighed flask, the naphtha distilled off by a water bath, and the residue weighed. From this weight the percentage of solubility shall be calculated.

FIXED CARBON

One gram of the bituminous material shall be placed in a platinum crucible, weighing between 20 and 30 grams, between 28 and 38 mm. (1.102 and 1.496 in.) in height, and having a tightly fitting cover provided with a flange, about 4 mm. (0.157 in.) in depth. The crucible and its contents shall then be heated, first gently and then more severely, until no smoke or flame shall issue between the crucible and the lid. It shall then be placed in the full flame of a bunsen burner for 7 min., holding the cover down with the end of a pair of tongs until the most volatile products shall have been burned off. The crucible shall be supported on a platinum triangle with the bottom from 6 to 8 cm. (2.362 to 3.150 in.) above the top of the burner. The flame shall be fully 20 cm. (7.874 in.) high when burning free, and the determination shall be made in a place free from drafts. The upper surface of the cover shall burn clear, but the under surface may or may not be covered with carbon, dependent on the character of the bituminous material. The crucible shall be removed to the desiccator, and, when cool, shall be weighed, after which the cover shall be removed and the crucible placed in an inclined position over the bunsen burner and ignited until nothing but ash remains. Any carbon deposited on the cover shall also be burned off. The weight of ash remaining shall be deducted from the weight of the residue after the first ignition of the sample. The resulting weight is that of the fixed carbon, which shall be calculated on the basis of the total weight of the sample, exclusive of mineral matter.

PARAFFIN

One hundred grams of the material shall be distilled rapidly in a retort to a dry coke. Five grams of the distillate shall then be thoroughly mixed in a 60-c.c. (2.029-oz.) flask with 25 c.c. (0.845 oz.) of Squibbs' absolute ether. Twenty-five c. c. (0.845 oz.) of Squibbs' absolute alcohol shall then be added, and the flask packed closely in a freezing mixture of finely crushed ice and salt for at least 30 min. The precipitate shall be filtered out quickly with a suction pump, using a No. 575 C. S. and S. 9-cm. hardened filter paper. The flask and precipitate shall then be rinsed and washed with a mixture of equal parts of Squibbs' alcohol and ether cooled to −17°C. (1°F.) until free from oil (50 cu. cm. (1.691 oz.) of washing solution is usually sufficient). When sucked dry, the filter paper shall be removed and the waxy precipitate transferred to a small glass disk and evaporated on a steam bath. The residue (paraffin) remaining on the disk shall be weighed, and from this weight the percentage on the original 5-gram sample shall be calculated.

SPECIFIC GRAVITY AT 38°C. OF WOOD BLOCK PRESERVATIVE[1]

A standardized hydrometer shall be used. A set of two with ranges 1.00 to 1.08, and 1.07 to 1.15 will suffice. Before taking the specific gravity, the oil in the cylinder should be stirred thoroughly with a glass rod, and this rod when withdrawn from the liquid should show no solid particles at the instant of withdrawal. Care should be taken that the hydrometer does not touch the sides or bottom of the cylinder when the reading is taken, and that the oil surface is free from froth and bubbles. If the specific gravity is determined at a higher temperature than desired, correction should be made by adding 0.0008 to the reading for each degree centigrade excess of temperature.

SOLUBILITY IN BENZOL OR CHLOROFORM OF WOOD BLOCK PRESERVATIVE

From 5 to 10 grams of the water-free oil is weighed out into a weighed 100-c.c. (3.38-oz.) beaker; 50 c.c. (1.69 oz.) of the solvent is added, and the solution is passed through a weighted 9-cm., C. S. and S., No. 575 filter paper in a short-stemmed funnel, the filtrate being passed into the flask to be subsequently used for the hot extraction. The beaker is washed clean from all soluble matter, dried, and weighed. The funnel, with filter paper and contents, is then placed in a N. Y. T. L. or Underwriter's form of glass extraction apparatus, and heat is applied from a water-bath or hot plate until the extraction is complete and the filtrate runs through colorless. The filter and contents are then dried and weighed. The increase in weight is added to the increase in weight of the beaker, if any, the result being the weight of the insoluble matter. The weight of the insoluble matter thus found is subtracted from the weight of the material taken for analysis. The difference in weight is the weight of the soluble matter, from which the percentage is calculated.

WATER CONTENT OF WOOD BLOCK PRESERVATIVE

From 250 to 300 c.c. (8.45 to 10.14 oz.) of the oil is weighed out into a 500-c.c. glass retort, or into a small copper still, provided with a distilling head. Heat is applied with a ring burner, starting with a small flame at the top of the still, and gradually lowering it until all the water has been driven off. The distillate of oil and water is collected in a graduated separatory funnel, the volume of water, in cubic centimeters, is read, and its percentage computed by volume. The water is then drawn off and the oils are returned to the residue in the still. The contents of the still shall have cooled to below 100°C. (212°F.) before the oils are returned, and they shall be well stirred and mixed with the residue.

DISTILLATION TEST FOR WOOD BLOCK PRESERVATIVE[2]

Apparatus for Distillation Test

Retort.—This shall be a tubulated Jena glass retort of the usual form, with a capacity of 250 to 290 c.c. (8.45 to 9.8 oz.). The capacity shall be measured by placing the retort with the bottom of the bulb and the end of the offtake in the same horizontal plane, and pouring water into the bulb through the tubulature until it overflows the offtake. The quantity remaining in the bulb shall be considered as its capacity.

Shield.—An asbestos shield shall be used to protect the retort from air currents and to prevent radiation. This may be covered with galvanized iron, as such an arrangement is more convenient and more permanent.

Receivers.—Erlenmeyer flasks of from 50 to 100 c.c. capacity are of the most convenient form.

Thermometer.—The thermometer shall be of glass, well annealed, and shall undergo no serious change at the zero point when heated up to 400°C. The space above the mercury column shall be filled with gas, either carbon dioxide or nitrogen, and the thermometer shall have an expansion chamber at the top. The scale shall read from 0 to 400°C., in graduations of 1°C., which shall be etched on the stem. The tip of the thermometer shall carry a ring for the purpose of attaching tags. The thermometer shall have the following dimensions:

[1] Modification of method proposed in 1915 by Committee D-7 on "Standard Specifications for Timber," of the Am. Soc. for Testing Materials.

[2] Modification of method proposed in 1915 by Committee D-7 on "Standard Specifications for Timber," of the Am. Soc. for Testing Materials.

Total length, 375 mm.; tolerance, 10 mm.
Bulb length, 14 mm.; tolerance, 1 mm.
Distance from zero mark to bottom of bulb, 30 mm.; tolerance, 4 mm.
Scale length from zero mark to 400°C., 295 mm.; tolerance, 5 mm.
Diameter of stem, 7 mm.; tolerance, 1 mm.
Diameter of bulb, 6 mm.; tolerance, 1 mm.
When standardized, the accuracy of such standardization should be as follows:

Up to 200°C.	to the nearest 0.5°C.
200 to 300°C.	to the nearest 1.0°C.
300 to 360°C.	to the nearest 1.5°C.

Assembling for Distillation Test

The retort shall be supported on a tripod or rings over two sheets of 20-mesh gage, 15.24 cm. (6 in.) square. It shall be connected to the condenser tube by a tight cork joint. The thermometer shall be inserted through a cork in the tubulature, with the bottom of the bulb 1.27 cm. (½ in.) from the surface of the oil in the retort. The exact location of the thermometer bulb shall be determined by placing a vertical rule, graduated in divisions not exceeding 0.16 cm. (1/16 in.), back of the retort when the latter is in position for the test, and sighting the level of the liquid and the point for the bottom of the thermometer bulb. The distance from the bulb of the thermometer to the outlet end of the condenser tube shall be not more than 60.96 cm. (24 in.) nor less than 50.8 cm. (20 in.). The burner should be protected from drafts by a suitable shield or chimney.

Distillation Test

Exactly 100 grams of oil shall be weighed into the retort, the apparatus shall be assembled and heat applied. The distillation shall be conducted at the rate of at least one drop, and not more than two drops, per second, and the distillate collected in weighed receivers. The condenser tube shall be warmed whenever necessary, to prevent accumulation of solid distillates. Fractions shall be collected at the following points:

Up to 170°C. (338°F.), 170–200°C. (338–392°F.), 200–210°C. (392–410°F.), 210–235°C. (410–455°F.), 235–270°C. (455–518°F.), 270–300°C. (518–572°F.), 300–315°C. (572–599°F.), 315–355°C. (599–671°F.).

The receivers shall be changed as the mercury passes the dividing temperature for each fraction. The last receiver shall be removed at 355°C. (671°F.), and drainage from the condenser, etc., shall not be considered as part of the fraction. For weighing the receivers and fractions, a balance accurate to at least 0.05 gram shall be used. During the progress of the distillation the thermometer shall remain in its original position. No correction shall be made for the emergent stem of the thermometer.

When any measurable quantity of water is present in the distillate, it shall be separated as nearly as possible and reported separately, all results being calculated on a basis of dry oil. When more than 2 per cent. of water is present, water-free oil shall be obtained by separately distilling a larger quantity, returning any oil carried over with the water, and using dried oil for the final distillation. A copper tar still is a convenient implement for obtaining water-free oil.

DISCUSSION

E. Dow Gilman,[1] Assoc. M. Am. Soc. C. E. (by letter).—Referring to page 470, on the subject "Abrasion Test for Broken Stone or Broken Slag," the writer notes that the specifications for this test as adopted in 1908 by the American Society for Testing Materials have been adopted in the report of the Committee. These specifications provide that the sample to be tested shall consist, as nearly as possible, of 50 pieces of the broken stone, the total weight of which shall be within 10 grams of 5 kilograms. Under the conditions thus stated, the sample may consist of broken stone the individual pieces of which may be of widely varying size and weight. That a sample composed of 50 pieces of nearly uniform size will give different results from those obtained with a sample composed of 50 pieces some of which are small and others large would seem to require no proof.

[1] Washington, D. C.

Several years ago the writer made abrasion tests on a shipment of trap rock. An attempt was made to check results by duplicating the tests on other samples taken from the same shipment. Three separate tests were made on as many different samples, each of which was carefully prepared according to the specifications just stated. Such varying results were secured that the writer was not satisfied to submit any one or the average of the French coefficients thus obtained. New samples were prepared, each consisting of 50 pieces of the broken stone and with the added specification that no single piece should weigh less than 90 or more than 110 grams, and that any piece of a shape noticeably different from the general shape of the entire lot should be eliminated. The total weight of the sample was within 10 grams of 5 kilograms, as before. The results were remarkably more consistent than with the other samples, and were so close together that, considering the unavoidable factors in a test of this nature, one would be warranted in submitting an average as the probable coefficient of the material at hand.

The objection to this added clause is the care required to prepare a sample and the quantity of material required from which to take one. In spite of this objection, the writer is inclined to believe that, if results secured by the abrasion test have any practical significance whatever, the test must be worthy of the slight extra labor involved. It seems to be a question whether the operation of the Deval machine for more than 5 hours and the labor in washing and drying the sample before and after the test are worth the time and energy, if the results of two or more samples of the same material are not reasonably close. Even with the utmost care, the tests for abrasion, hardness, toughness, and cementing value, give results that are rough and that can be taken as no more than a preliminary guide in passing judgment on the usefulness of a material. With the limited use that at present is made of the results of these tests, it would be unwise to burden them with too many refinements, but the writer submits the suggestion here made in the belief that greater reliance can be placed on comparative results thus obtained. This fact is apparently acknowledged in the specifications for the abrasion test of gravel, immediately following, which, under Method No. 2, insures, at least to a degree, the uniformity of separate samples.

The writer regrets that the data secured in the tests which he made are not available, so that by actual figures he might show comparisons of the methods; but he believes that the point in question can be raised without these figures, and that the matter is worthy of confirmatory investigation, with possible modification in the outline of the test at some later date.

J. O. Preston,[1] Jun. Am. Soc. C. E. (by letter).—The Committee, on page 439, invites information on the subject of "proper methods of sampling highway materials." This work is usually done in the field by inspectors who may or may not have written laboratory instructions. The instructions usually convey to the inspector a notion of the need for an average sample. It has been the writer's experience that the "average" notion is much overworked.

Materials that vary between extreme limits over the work are said to average well. This is especially true with concrete materials. As a result, in many cases the variations in density of adjoining batches of concrete cause cracking, due to irregularities in the grading of the sand and stone, and the consequent widely varying coefficient of expansion. A composite sample of an aggregate from even a small area rarely indicates the actual conditions. If the average is uniform throughout the work, or if all the materials vary but slightly from the average, such a sample would be a fair specimen. In practice, however, this is seldom the case.

Experience has proved the inadequacy of leaving to field inspectors the determination of the quality of materials. For the same reason, it is inadvisable that the selection of samples from varying grades of materials, such as sand and stone, be left to the average inspector's "eye." The only safe procedure is to have trained men in the laboratory for the purpose of going into the field and collecting all samples.

It is believed that it will prove ultimate economy for State or municipal laboratories (and all other large-scale organizations), if instead of awaiting samples to be sent in for test, they develop an organization to collect them. This organization would prove of great value in many other ways; not only would laboratory control be more complete, but field inspectors could be instructed in the proper use of materials. (Engineers seldom have time for sufficient instruction in the field.) The slight additional cost would be cheap insurance; it would aid both the engineer and the contractor to obtain better materials and prevent their misuse or adulteration.

[1] Rochester, N. Y.

Prewar Railway Specifications for Petroleum Products

	Product	Bé. 60°F./60°F.	Flash	Fire	Cold	Color	Flash and fire apparatus	Date of specification	Other requirements
The Baltimore and Ohio Railroad Co.	Fuel oil	18°–37°	110°F.		10°F. Nov. 1–Mar. 1			Aug. 10, 1914	Must be a fraction of crude petroleum, or the crude itself. Viscosity 250 sec. Saybolt or less at 80°F. from Nov. 1–March 1, or 250 sec. Saybolt or less at 100°F. for remainder of year. B. S., 2 per cent.
	150° fire test oil	45°–48°	125°F.	150°F.	0°F.	Water white	Tagliabue open cup	Feb. 25, 1911	No flocking when heated to 270°F. for 1 hr. No sulphur. Lamp test, burn freely and steadily for 24 hrs. In standard long time burner, 12 oz. of oil must be entirely consumed.
	300° fire test oil	38°–42°	250°F.	300°F.	32°F.	Standard white	Tagliabue open cup	Feb. 25, 1911	Must not flock when heated to 450°F. Must burn freely and steadily with standard burners and wicks.
	Signal oil	34°–38°	250°F.	300°F.	17°F.			Feb. 5, 1910	Must contain at least 22 per cent. mineral oil. Lamp test, flame turned high as possible without smoking. Show not less than 1.4 c.p. Not more than 0.5 per cent. of free acid.
Santa Fe System	Headlight oil	42°–46°	110°F.			Water white	Elliott closed cup	May 31, 1902	Must be free from glue and other suspended matter.
	Mineral seal oil	32°–40°	249°F.	300°F.		Water white	Tagliabue open cup	May 1, 1908	Must be free from glue and other suspended matter.
	Fuel oil	13°–25°	110°F.				Tagliabue open cup	June 29, 1911	Must be sufficiently liquid to flow readily in pipes when heated to temperature of 60°F. Must be free from sticks, waste, stones, etc. B. S. 2 per cent.
	Petroleum asphalt							Dec. 12, 1912	Sp. gr. 0.95–1.0 at 60°F. Must be soluble in pure carbon disulphide to extent of 98 per cent. Less than 1 per cent. sulphur. Melting-point by ball and ring method, 170°F.–190°F.

Southern Pacific Railroad.	Gas oil	30°–35°	120°F. to 150°F.	140°F. to 175°F.			Tagliabue open cup	Mar. 9, 1918	Must not contain asphalt or sludge.
	Gas engine distillate	46°–60°				Water white		June 10, 1913	Free from water and sediment.
	Headlight oil	41°–48°	130°F.	150°F.	0°F. for 10 min.		Tagliabue open cup	Aug. 4, 1912	
	Mineral seal oil	33½°–43°	245°F.	300°F.	32°F. for 10 min.			Aug. 4, 1912	
	Gasoline	Not less than 56°						Aug. 4, 1912	A portion of sample must be entirely volatile at a temperature not exceeding 100°F. A few drops on blotting paper must evaporate entirely, leaving no greasy stain.
	Fuel oil	13°–29°	110°F.				Tagliabue open cup	Aug. 4, 1912	If containing more than 2 per cent. water and sediment, will not be accepted.
	Wiping distillate	Not less than 29°	110°F.					June 10, 1913	Approximate viscosity of kerosene oil. No offensive odor. Free from water and sediment.
Pennsylvania Railroad Co.	150°F. fire test		130°F.	151°F.	0°F. for 10 min.	Water white		May 25, 1900	No glue or suspended matter. Must give satisfactory flame for six days continuous burning in long time burning switch lamp; no appreciable hard crust formed.
	300°F. fire test		249°F.	298°F.	32°F. for 10 min.	Water white		May 25, 1900	No precipitation when slowly heated to 450°F.
	Paraffin and neutral oils	24°–35°	298°F.		10°F. winter; 32°F. summer			May 25, 1900	Not so dark in color that long primer printing cannot be read with ordinary daylight through layer of oil ½ in. thick.
	Well oil	28°–31°	See last column		10°F. winter; 32°F. summer			May 25, 1900	Must show no precipitation when 5 c.c. are mixed with 95 c.c. of gasoline. Flash point, May 1st to Oct. 1st, 298°F.; Oct. 1st to May 1st, 249°F.
	530°F. flash test	Not below 25°	522°F.					May 25, 1900	No precipitation with gasoline as under well oil.

RAILWAY SPECIFICATIONS FOR PETROLEUM PRODUCTS.—*Continued.*

	Product	Bé. $\frac{60°F.}{60°F.}$	Flash	Fire	Cold	Color	Flash and fire apparatus	Date of specification	Other requirements
The Wabash Railroad.	Gasoline					Water white		Jan. 4, 1909	Must not show residue on evaporating one ounce at 100°F. Must not show stain on white blotting paper after evaporating at 100°F.
	Benzine	58°–62°							
	Long time switch and signal oil		See last column	See last column				April 28, 1914	Lamp test in A. W. No. 63 B. S. switch lamp with flame ¾ in. neight; after 110 hrs. flame shall not be below ⅝ in. high. Must not smoke or form crust. The oil must be entirely consumed. Flash and fire—Ia., Mo., Ill., Ind., O., and Mich. state requirements.
	150° headlight oil	42°–45.5°	110°F. to 120°F.	150°F.	No cloud at 0°F. for 10 min.	Water white	Elliot c.c. and Tagliabue o.c.	Jan. 4, 1909	Must burn free without encrusting wick in standard lamps and burner, render satisfactory signal, and not cease to burn until entirely consumed.
	300°F. mineral seal	32.5°–38°	249°F. to 269°F.	300°F.	No cloud at 32°F. for 10 min.	Straw	Elliot c.c. and Tagliabue o.c.	Jan. 4, 1909	Must burn free without encrusting wick in standard lamps and burner, render satisfactory signal, and not cease to burn until entirely consumed.
	300° signal oil		249°F. to 274°F.	300°F.	No cloud at 32°F. for 10 min.	Straw		Jan. 4, 1909	Must burn free without encrusting wick in standard lamps and burner, render satisfactory signal, and not cease to burn until entirely consumed.
Union Pacific Railway Co.	Headlight oil	41°–48°	130°F.	150°F.	No cloud at 0°F. for 10 min.	Water white	Tagliabue open cup	April 1, 1914	Free from sulphur. Cracked oil not desired.

Union Pacific Railway Co.	Mineral seal	33.5°–43°	245°F.	300°F.	No cloud at 32°F. for 10 min.	Water white	Tagliabue open cup	April 1, 1914	Free from sulfur.
	Elaine oil for semaphore lamps	Not below 48°	125°F.	150°F.	No cloud at 0°F. for 10 min.	Water white	Elliot and Tagliabue	April 1, 1914	Must burn in company's standard semaphore lamp without smoking and minimum incrustation of wick for 120 hours.
	Eocine oil for switch lamps	Not below 42°	120°F.	155°F.	No cloud at 0°F. for 10 min.	Water white	Elliot and Tagliabue	April 1, 1914	Must burn in company's standard switch lamp for period at least 72 hours without smoking and with minimum incrustation of wick.
	Gasoline	Not below 60°						April 1, 1914	Portion must be entirely volatile at 100°F. Drops on blotting paper must evaporate, leaving no greasy stain.
	Fuel oil	13°–29°	110°F.				Tagliabue	April 1, 1914	Must be sufficiently liquid to flow readily in 4-in. pipes at temperature of 70°F. B. S. 2 per cent.
Canadian Pacific Railway Co.	Gasoline					Water white			When dropped on blotting paper must evaporate without leaving stain.
	Benzine					Water white			When dropped on blotting paper must evaporate without leaving stain.
	Headlight oil	44°–48°	95°F.		No cloud at 10°F. for 10 min.	Water white	Abel closed cup	July 16, 1909	No water, glue, or suspended matter.
	Switch and semaphore lamp oil	46°–48°	90°F.		No cloud at 30°F. for 10 min.	Water white	Abel closed cup	July 16, 1909	No water, glue, or suspended matter.
	Transformer oil							June 24, 1910	Transformers and starting boxes. Shall not contain moisture exceeding 0.5 per cent.; and where voltage exceeds 10,000, moisture shall not exceed 0.05 per cent.
Chicago, Milwaukee and St. Paul Railway Co.	Headlight	42°–48°	110°F.			Water white	Elliot closed cup	Mar. 21, 1912	Cold tests received between April and Oct.—no cloud at 0°F. for 30 min. Cold tests received between Oct. and April—no cloud at 20°F. for 30 min.

RAILWAY SPECIFICATIONS FOR PETROLEUM PRODUCTS.—*Concluded.*

	Product	Bé. 60°F./60°F.	Flash	Fire	Cold	Color	Flash and fire apparatus	Date of specification	Other requirements
Chicago, Milwaukee and St. Paul Railway Co.	Mineral seal	33°–42°	235°F.		No cloud at 32°F. for 30 min.	Practically white	Elliott closed cup		
	Cylinder stock	Not below 25°	Not below 475°						Known as dark filtered. No coke or tar. When churned with water at 212°F. for 12 hrs., oil must not thicken or turn brown.
	Fuel for G. E. motor cars								Initial boiling point not above 185°F. (85°C.). Dry point not in excess 550°F. (288°C.). First 10 per cent. should not distil above 230°F.—60 per cent. should not distil above 374°F.
	Fuel oil		110°F.				Elliott closed cup	June 11, 1900	Melting-point in winter not higher than 25°F. or 40°F. in summer. B. S. 1 per cent.

Specifications for Transformer Oil as Adopted by the Railway Signal Association

1. Material.—Shall be a pure mineral oil obtained by the fractional distillation of petroleum, unmixed with any other substance. It shall be prepared and refined especially for the purpose and shall be free from moisture, acid and alkali, and shall not contain sulphur compounds to exceed one-fifth of one per cent.

2. Tests.—(*a*) The flash of the oil determined in an open cup shall be not less than three hundred and twenty (320) degrees Fahr. (160°C.) and on fire test not less than three hundred and forty-seven (347) degrees Fahr. (175°C.).

(*b*) The viscosity shall be not more than sixty (60) seconds at one hundred and four (104) degrees Fahr. (40°C.) by the Saybolt universal viscosimeter.

(*c*) The oil shall stand a breakdown test of thirty thousand (30,000) volts a.c. between spherical surfaces of 0.5 cm. radius, 0.40 cm. apart.

A Resumé of the Investigation of Campbell and Wilson on the Sweating of Crude Paraffin Wax. (From J. Inst. Pet. Tech., 5, No. 18, 106.)

The objects of the investigation were:

(*a*) To observe the effect of variation of time and rate of temperature-increase of sweating.

(*b*) To ascertain the advantage or disadvantage of combining the scales obtained by separate filtrations at different temperatures, as compared with the separate sweating of the crude products.

The scale experimented with was extracted from the heavy oil and paraffin distillates in two stages, the hard scale melting at 129°F., and the soft scale, extracted at 32°F., at 113°F. Both were well pressed, and contained only a very small percentage of oil. When mixed together in the proportions in which they were obtained, the average melting-point of the mixture was 120.5°F.

The finished products aimed at were paraffins of 135° and 125° melting-point, respectively, both to be sufficiently refined for the final treatment of filtration through decolorizing earth, and to yield a perfectly white product. Generally, the melting-points of the fractions obtained varied from these standards, but for the purpose of comparison in compiling the results tabulated below the quantities obtained have been adjusted by calculation.

The scales in each case were melted in a shallow tray of suitable size, and allowed to cool slowly to atmospheric temperature. The cake of scale was transferred to a tray of interlaced wire, the bottom of which was covered by a fine gauze. The wire tray was placed in a sloping position in a larger metal tray in which the sweatings were received. The whole was contained in a large water-oven fitted with a thermo-regulator, by means of which the temperature was maintained within about 1°F. of the temperature required at each stage of the operations. At the end of each stage, the sweatings were removed and weighed and their melting-points determined. The quantity of scale dealt with in each experiment was 500–600 g. The diagrams A, B, C and D indicate the products actually obtained, and also the adjustments necessary to give the exact melting-points required.

DIAGRAM A

Total Scale = 100 per cent.

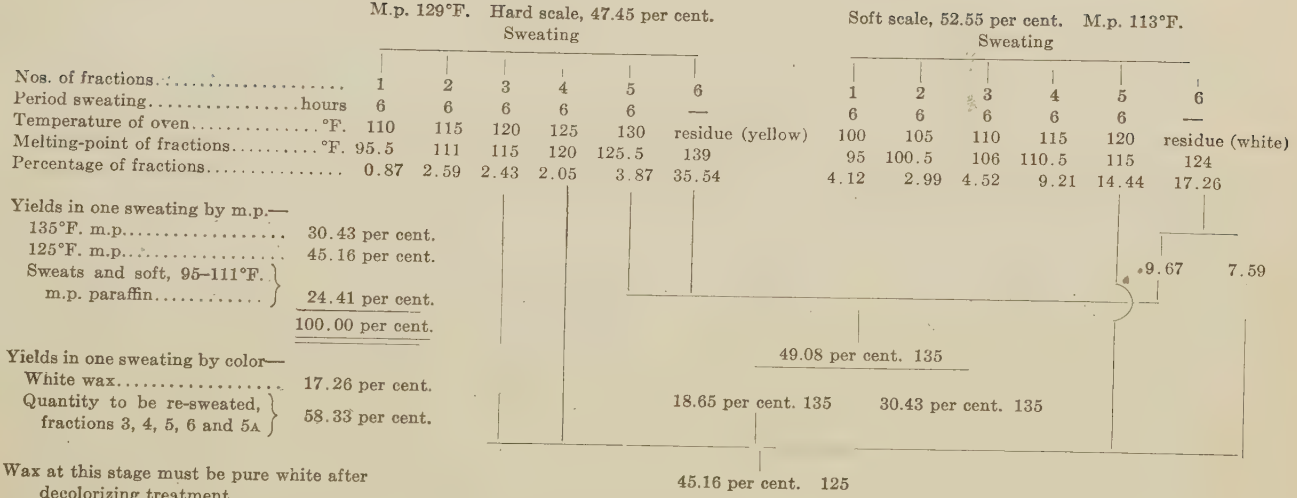

M.p. 129°F. Hard scale, 47.45 per cent.
Sweating
Soft scale, 52.55 per cent. M.p. 113°F.
Sweating
Nos. of fractions 1 2 3 4 5 6 1 2 3 4 5 6
Period sweating....hours 6 6 6 6 6 — 6 6 6 6 6 —
Temperature of oven....°F. 110 115 120 125 130 residue (yellow) 100 105 110 115 120 residue (white)
Melting-point of fractions....°F. 95.5 111 115 120 125.5 139 95 100.5 106 110.5 115 124
Percentage of fractions.... 0.87 2.59 2.43 2.05 3.87 35.54 4.12 2.99 4.52 9.21 14.44 17.26
Yields in one sweating by m.p.—
135°F. m.p.... 30.43 per cent.
125°F. m.p.... 45.16 per cent.
Sweats and soft, 95–111°F. m.p. paraffin.... 24.41 per cent.
100.00 per cent.
Yields in one sweating by color—
White wax.... 17.26 per cent.
Quantity to be re-sweated, fractions 3, 4, 5, 6 and 5A 58.33 per cent.
Wax at this stage must be pure white after decolorizing treatment.
9.67
7.59
49.08 per cent. 135
18.65 per cent. 135
30.43 per cent. 135
45.16 per cent. 125

DIAGRAM B

Total Scale = 100 per cent.—M.p. 120.5°F.

Sweating

A. No. of fractions	1	2	3	4	5	6	7	8
B. Period of sweating.....hours	6	6	6	6	6	6	6	—
C. Temp. of oven.....°F.	100	105	110	115	120	125	130	residue (white)
D. M.p. of fractions.....°F.	89	101.5	104	110.5	116	121	124.5	135.5
E. Percentage of fractions	4.07	5.55	6.13	5.46	7.83	12.37	16.78	41.80

Re-sweating 42.44 per cent.

A.	1	2	3	4
B.	6	6	6	—
C.	105	110	115	residue
D.	100.5	106	113	125 (white)
E.	4.39	2.80	3.30	31.97

29.88 2.09
125

43.89
1.35

Yields in two sweatings by m.p.—

135°F. m.p.	43.89 per cent.
125°F. m.p.	29.88 per cent.
Sweats and soft paraffin, 89–113°F. m.p.	26.23 per cent.
	100.00 per cent.

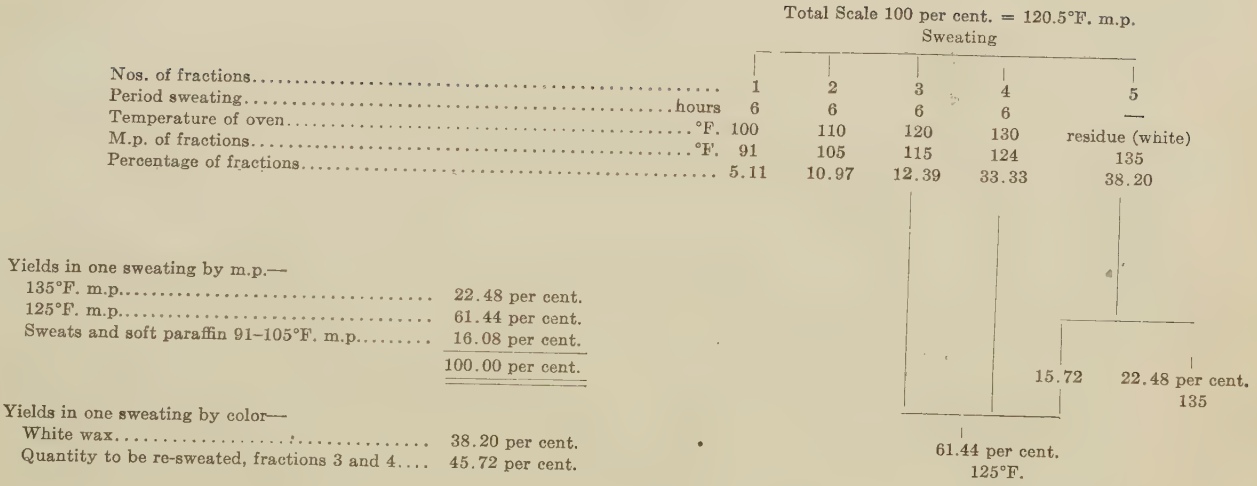
Yields in one sweating by color—

White wax	41.80 per cent.
Quantity to be re-sweated,— fractions 4–7	42.44 per cent.

Re-sweating Soft Fractions

A.	1	2	3	4	5	6	7
B.	6	6	6	6	6	6	residue (yellow)
C.	90	95	100	105	110	115	—
D.	84	92	97	101.5	107.5	112.5	122.5
E.	11.10	1.35	1.13	1.11	1.52	3.50	6.53

DIAGRAM C


Total Scale 100 per cent. = 120.5°F. m.p.
Sweating
Nos. of fractions 1 2 3 4 5
Period sweating ..... hours 6 6 6 6 —
Temperature of oven ..... °F. 100 110 120 130 residue (white)
M.p. of fractions ..... °F. 91 105 115 124 135
Percentage of fractions 5.11 10.97 12.39 33.33 38.20
15.72
22.48 per cent.
135
61.44 per cent.
125°F.

Yields in one sweating by m.p.—

135°F. m.p.	22.48 per cent.
125°F. m.p.	61.44 per cent.
Sweats and soft paraffin 91–105°F. m.p.	16.08 per cent.
	100.00 per cent.

Yields in one sweating by color—

White wax	38.20 per cent.
Quantity to be re-sweated, fractions 3 and 4	45.72 per cent.

DIAGRAM D

Total Scale 100 per cent. = 120.5°F. m.p.

	Sweating				
Nos. of fractions	1	2	3	4	5
Period sweating.....hours	4	4	4	4	—
Temperature of oven.....°F.	100	110	120	130	residue
Melting-point of fractions.....°F.	91.5	106	115	124.5	135
Percentage of fractions	3.11	13.02	14.26	31.93	37.67

Yields in one sweating by m.p.—

135°F. m.p.	21.81 per cent.
125°F. m.p.	62.05 per cent.
Sweats and soft paraffin, 9.15–106	16.14 per cent.
	100.00 per cent.

15.86 per cent. 21.81 per cent.
135

62.05 per cent.
125

Yields in one sweating by color—

White wax	37.67 per cent.
Quantity to be re-sweated, fractions 3–4	46.19 per cent.

Reference to the diagrams will show that in experiment A, when the hard and soft scales were sweated separately, only 17.26 per cent. from the latter reached the proper color, and that 58.33 per cent. was still yellow, and required re-sweating to obtain the maximum yield of white wax.

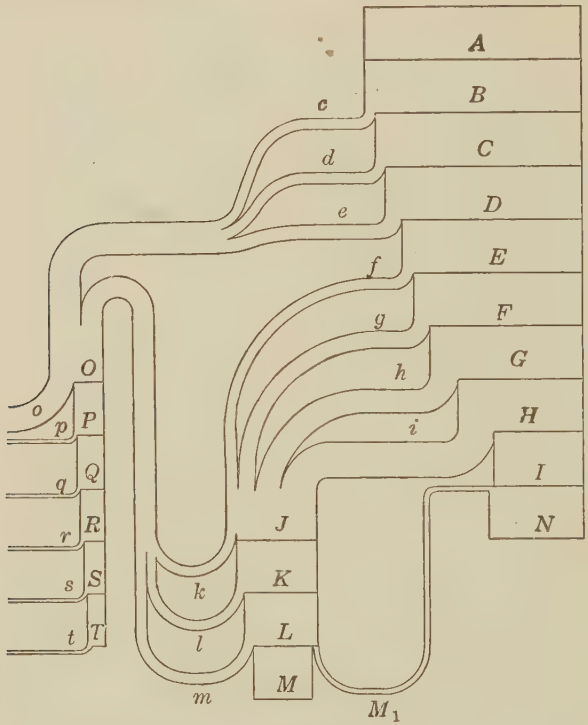


FIG. 136.—Sweating of paraffin scale (temperature increased 5°F. every 6 hrs.), according to Campbell and Wilson.

FIRST STAGE SWEATING

A, Total Scale, 100 per cent.; M.P., 120.5°F.: B, Stove Temperature, 100°F.: C, 105°F.: D, 110°F.: E, 115°F.: F, 120°F.: G, 125°F.: H, 130°F.: I, Residue White Wax from First Stage Sweating, 41.80 per cent.; M.P., 135.5°F. c–i, Sweats from First Stage Sweating. c, 4.07 per cent.; M.P., 89°F.: d, 55.55 per cent.; M.P., 101.5°F.: e, 6.13 per cent.; M.P., 104°F.: f, 5.46 per cent.; M.P., 110.5°F.: g, 7.83 per cent.; M.P., 116°F.: h, 12.37 per cent.; M.P., 121°F.: i, 16.78 per cent.; M.P., 124.5°F.

MIDDLE SWEATS (f, g, h, i= 42.44 per cent.), CRYSTALLIZED AND RESWEATED.

J, Stove Temperature, 105°F.: K, 110°F.: L, 115°F.: M + M_1, Residue White Wax from Second Stage Sweating, 29.88 per cent. = 2.09 per cent. + 31.97 per cent.; M.P., 125°F.

k–m, Sweats from Second Stage Sweating. k, 4.39 per cent.; M.P., 100.5°F.: l, 2.80 per cent.; M.P., 106°F.: m, 3.30 per cent., M.P., 113°F.

SOFT SWEATS (c, d, e, k, l, m = 26.24 per cent.), CRYSTALLIZED AND RESWEATED.

O, Stove Temperature, 90°F.: P, 95°F.: Q, 100°F.: R, 105°F.: S, 110°F.: T, 115°F.

Residue Wax (colored) from Third Stage Sweating, 6.53 per cent.; M.P., 122.5°F.

o–t, OILY SWEATS, 19.70 PER CENT. FROM THIRD STAGE SWEATING RETURNED TO PROCESS OR BURNED AS FUEL.

o, 11.10 per cent.; M.P., 84°F.: p, 1.35 per cent.; M.P., 92°F.: q, 1.13 per cent.; M.P., 97°F.: r, 1.11 per cent.; M.P., 101.5°F.: s, 1.52 per cent.; M.P., 107.5°F.: t, 3.50 per cent.; M.P., 112.5°F.

WHITE WAX. I + M_1 = 43.89 per cent.; M.P., 135°F.

Experiment B, in which the scales were combined proportionately, yielded no less than 41.8 per cent. white in one operation, and only 42.24 per cent.

required re-sweating. When this was done a further quantity, practically 32 per cent., reached the white stage.

As will be observed, these experiments were carried out in stages of 6 hours, sweating at each rise of 5°F. in the temperature of the oven. By comparison of the time occupied in each case, 30 hours sweating in A yielded only 41.2 per cent. of the white product yielded in 42 hours in B.

That is to say, with an apparatus containing 100 per cent. of scale divided in two parts as in A, 30 hours were taken to sweat the residues to 135.5°F. and 125°F. respectively—the latter only being white—while the same size of apparatus working the same quantity of scale as in B produced more than double the yield of white wax in 42 hours.

The authors then proceeded to ascertain if a similar result was obtainable in a shorter period. Diagrams C and D show that when the periods were reduced to 6 hours, and ultimately 4 hours' duration, with rises of 10°F., yields similar to those in B were obtained. They had therefore succeeded in reducing the total period for satisfactory sweating to 16 hours, as compared with the unsatisfactory treatment of the scales separately lasting 30 hours. Consequently, considering all these results conjointly, with the apparatus described could be obtained similar results to the lengthy sweating in B, by working the first stage as in D for 16 hours, and the second stage as in B for 18 hours.

It will be seen from diagram B that when the sweatings from the first and second stages were mixed, crystallized, and again sweated, 6.5 per cent. melting at 122.5°F. was recovered, although not white in color. The fractions sweated out at this stage were more or less oily. The following are the conclusions to be drawn from these experiments.

1. Sweating of the total scale produced a much greater yield of white paraffin in less time than the treatment of the hard and soft scales separately. While the authors recognize the necessity for special experiment with the scale produced from different crude oils, they are inclined to think that the same result will obtain generally, provided the distillates are of good quality, and the melting-point range of the scale similar to that used in these experiments.

2. The efficiency of the fractional sweating depends on the efficiency of the apparatus employed. One type of apparatus, worked by both the above methods, will bear the same relation to each other as the experiments, and give comparative results.

Analyses of Natural Gas Collected in 31 Cities in the United States. (From Bureau of Mines Tech. Paper 158.)

Laboratory No.	State	County	Town	Paraffin hydrocarbons (C_nH_{2n+2})	Methane (CH_4)	Ethane (C_2H_6)	Carbon dioxide (CO_2)	Nitrogen (N_2)	Calculated gross heating value, B.t.u. per cubic foot (760 mm. pressure)		Calculated specific gravity (air = 1)
									0°C.	60°F.	
7176	Ala.	Fayette	Fayette	97.6	97.6	0.0	0.3	2.10	1,039	983	0.57
7309	Ark.	Crawford	Alma	99.2	99.2	0.0	0.20	0.6	1,057	1,000	0.56
6888	do	Pulaski	Little Rock	96.7	96.7	0.0	1.00	2.3	1,030	974	0.57
7341	Cal.	Los Angeles	Los Angeles	93.5	77.5	16.0	6.50	0.0	1,123	1,062	0.70
7359	do	do	do	73.1	59.2	13.9	26.20	0.7	889	841	0.88
7340	do	do	do	98.0	70.0	28.0	1.10	0.9	1,267	1,199	0.71
7039	Ill.	Lawrence	Olney	97.1	37.5	59.6	0.0	a1.7	1,591	1,505	0.86
6857	do	Crawford	Palestine	95.6	95.6	0.0	0.5	3.9	1,018	963	0.58
7147	Ind.	Adams	Geneva	98.8	75.4	23.4	0.0	1.2	1,238	1,171	0.68
7280	Kans.	Montgomery	Coffeyville	98.0	98.0	0.0	1.2	0.8	1,044	988	0.57
7148	do	Crawford	Pittsburg	93.0	90.5	2.5	0.4	6.6	1,010	955	0.60
7173	Ky.	Boyd	Ashland	79.0	75.0	24.0	0.0	1.0	1,245	1,178	0.68
7265	do	Fayette	Lexington	99.0	76.4	22.6	0.0	1.0	1,234	1,167	0.67
6847	Mo.	Jackson	Kansas City	90.8	84.1	6.7	0.8	8.4	1,025	965	0.63
7233	N. Y.	Chenango	Elmira	99.0	84.0	15.0	0.0	1.0	1,174	1,111	0.63
7240	do	Allegany	Bolivar	97.4	59.8	37.6	0.4	2.2	1,336	1,264	0.75
6853	do	Erie	Buffalo	99.6	88.1	11.5	0.0	0.4	1,152	1,090	0.61
6856	do	Genesee	Pavilion	98.7	91.9	6.8	0.0	1.3	1,105	1,045	0.59
6817	do	Allegany	Wellsville	98.0	78.1	19.9	0.0	2.0	1,202	1,137	0.65
7151	Ohio	Ashtabula	Ashtabula	98.7	82.2	16.5	0.0	1.3	1,182	1,118	0.65
7357	do	Allen	Lima	96.3	83.5	12.8	0.0	3.7	1,127	1,066	0.63
7169	do	Miami	Piqua	90.9	78.3	12.6	0.2	8.9	1,068	1,010	0.66
7171	do	Erie	Sandusky	96.0	83.5	12.5	0.2	3.8	1,122	1,061	0.63
7266	do	Licking	Utica	93.9	74.8	19.1	0.3	5.8	1,152	1,090	0.68
7279	Okla.	Logan	Guthrie	91.9	73.5	18.4	0.0	8.1	1,125	1,064	0.68
7277	do	Creek	Sapulpa	98.8	93.1	5.7	0.4	0.8	1,098	1,039	0.59
7181	Pa.	Blair	Altoona	99.0	99.0	9.0	0.2	0.8	1,126	1,065	0.60
6815	do	Venango	Oil City	97.7	64.3	33.4	0.0	3.3	1,306	1,235	0.74
7353	do	McKean	St. Marys	99.2	88.0	11.2	0.0	0.8	1,146	1,084	0.61
7167	do	do	Sharon	99.3	32.3	67.0	0.0	0.7	1,591	1,505	0.89
6845	W. Va.	Kanawha	Charleston	99.3	76.8	22.5	0.0	0.7	1,236	1,169	0.67
7384	do	Harrison	Clarksburg	99.3	66.6	32.7	0.0	0.7	1,318	1,247	0.72
7175	do	Marion	Fairmont	99.0	82.0	17.0	0.1	0.9	1,189	1,125	0.64

a Contained also 1.2 per cent. hydrogen sulphide (H_2S).

NOTE.—The sum of the methane, ethane, carbon dioxide and nitrogen totals 100 per cent.

The Properties of Seven Paraffin Hydrocarbons. (Bureau of Mines Tech. Paper 88)

Hydrocarbon	Formula	Boiling point *b*, °C.	Specific gravity (at 0°C. and 760 mm.; air = 1)	Weight of 1 liter, grams	Heating value per cubic foot at 0°C. and 760 mm. *c*, B.t.u.	Illuminating value, British candle-power	Liquefaction point, lb. per sq. in. °C.	Calculated volume of gas (at 60°F. and 30 in. barometer) pressure from 1 gal.	Theoretical volume of air necessary to burn 1 cu. ft. of gas, cu. ft.
Methane *b*	CH_4	−160	0.554	0.7159	1,065	5.0	−95.5 at 735 *f* −81.8 at 807 *g*	..	9.57
Ethane *d*	C_2H_6	− 93	1.049	1.3567	1,861	*h* 53.0	+35 at 664 *i*	53	16.72
Propane *d*	C_3H_8	− 45	1.520	1.9660	2,654	*h* 53.9	+97 at 647 *g*	45	23.92
Butane *d*	C_4H_{10}	1.0	2.004	2.5940	3,447	....	.....	37	31.10
Pentane *k*	C_5H_{12}	36.4	.....	.....	4,250	....	.....	31	38.28
Hexane *k*	C_6H_{14}	68.9	.....	.....	5,012	....	.....	27	
Heptane *k*	C_7H_{16}	98.4							

b HOLLEMAN, A. F., "Organic Chemistry," edited by A. J. WALKER, **1910,** p. 41.
c LANDOLT and BÖRNSTEIN, "Physikalisch-chemische Tabellen," 3d ed., **1905,** pp. 416, 425 (J. THOMPSON).
d Gas at ordinary temperature.
e WRIGHT, L. T., Illuminating power of methane, *J. Chem. Soc.*, **47** (1885), 200.
f LANDOLT and BÖRNSTEIN, "Physikalisch-chemische Tabellen," 3d ed., **1905,** p. 185 (Dewar).
g LANDOLT and BÖRNSTEIN, "Physikalisch-chemische Tabellen," 3d ed., **1905,** p. 185 (Olszewski).
h FRANKLAND, P., Illuminating power of methane, *J. Chem. Soc.*, **47** (1885), 235.
i LANDOLT and BÖRNSTEIN, "Physikalisch-chemische Tabellen," 3d ed., **1905,** p. 182 (Dewar).
k Liquid at ordinary temperature.

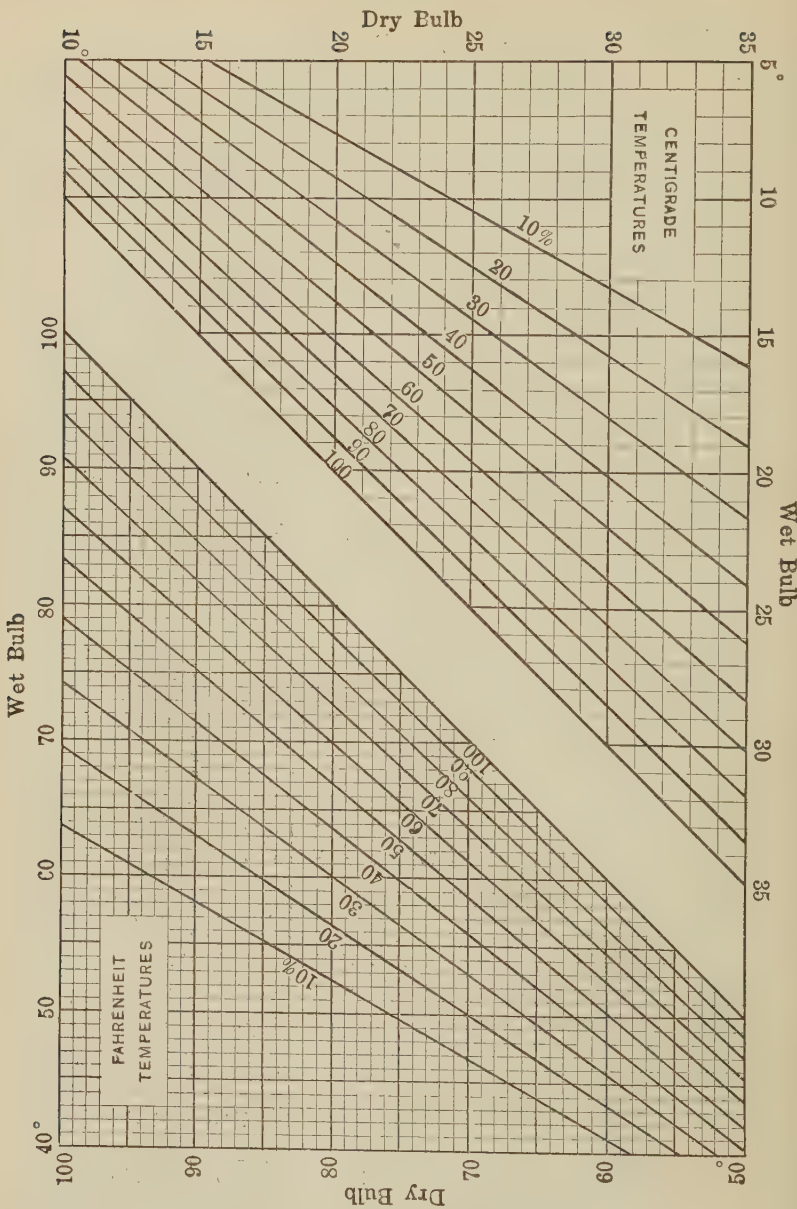


FIG. 137.—Relative humidity from wet and dry bulb thermometer readings for psychrometer with rapid ventilation. Calculated for a barometer height of 755 mm. At altitudes of 6000 ft. or over the reduction in barometric pressure will cause an error of 5 per cent. or over at very low humidities.

Limits of Explosibility of Gases

	Low limit	High limit
Pittsburgh natural gas	5.0 per cent. gas	11.6 per cent. gas
Casinghead	3.5 per cent. gas	9.5 per cent. gas
Gasoline vapor	1.4 per cent. gas	6.0 per cent. gas

Calorimetric Laboratory and Equipment. (From Bureau of Standards Circular 48.)

(*a*) **Laboratory.**—If a number of rooms are available, one should be chosen the temperature of which is not subject to rapid changes during the times at which observations will be made; a room which is not subjected to direct sunlight at about the time observations are to be made is therefore desirable. A room used exclusively for the calorimetric or calorimetric and photometric laboratory is to be preferred.

Connections to a water supply and to the supply of gas to be tested must of course be available in the laboratory, and it is a great convenience also to have a hot-water supply. A sink with water and drain connections should be installed in the laboratory. The gas-supply pipe leading from the main to the laboratory should be suitably located and properly drained; precautions to be observed in obtaining gas for testing purposes are given on page 506.

(*b*) **Equipment.**—If a flow calorimeter of the ordinary type is to be installed, the following equipment for the calorimetric laboratory will be required, in addition to the necessary piping (for hot and cold water, gas, and drain), valves, cocks, hose nipples, tubing for connections, etc.

1. Calorimeter and accessories.
2. Thermometers, reading glasses.
3. Apparatus for weighing or measuring water.
4. Small graduate for measuring condensate.
5. Gas meter.
6. Pressure regulators.
7. U-gage for measuring gas pressure.
8. Barometer.
9. Psychrometer.
10. Gas holder (not always necessary).
11. Overhead water tank. } or (13.1) Calorimeter cabinet.
12. Table. }

Apparatus for calibrating the meter by one of the methods described on page 532 is a very desirable although not an absolutely necessary part of the equipment.

1. *Calorimeter.*—The choice of calorimetric apparatus is discussed in *Circular* **48** of the Bureau of Standards.

2. *Thermometers.*—The inlet and outlet water thermometers should be graduated to 0.1° or 0.2°F.

At least four calorimetric thermometers should be provided for each calorimeter to avoid delays otherwise resulting from breakage of thermometers. Detailed specifications for high-grade calorimetric as well as for other laboratory thermometers will be published in a special Bureau circular now in preparation.

For convenience in reading the calorimetric thermometers and to avoid errors due to parallax, suitable reading glasses conveniently movable along the stems of the thermometers should be provided.

Thermometers graduated to 1°F. are suitable for measuring the temperature of the gas and the products of combustion.

3. *Apparatus for Weighing or Measuring Water.*—It is easily possible to weigh the water with an accuracy beyond that required for calorimetric work, or, with suitable precautions, to measure the water volumetrically with sufficient accuracy.

The scale should be of such construction that weighings can be conveniently and quickly made. The combined errors of the scale (such as those due to inequality of arms, to varying positions of the weights on the scale pan, limiting sensibility due to friction, etc.) and of the weights should be so small that the masses of water to be weighed in calorimetric tests (about 7 lb.) can be determined with an accuracy of 1 part in 1000. The buckets in which the water is collected during a test should all be of the same weight, and the scale should preferably be so made that the weighing bucket is exactly counterpoised when in position on the scale. The weights, rider, and graduation of the arm of the scale should be such that the weighings are made directly in pounds and decimals of a pound.

Graduates of cylindrical form are often used to measure the volume of water collected in calorimetric tests. While it is possible to use such graduates with an accuracy of 0.2 per cent., this requires so much time and care in reading that the main advantages in measuring the water over weighing it are lost. It is preferable to use a graduate in which the lower portion is enlarged and only the upper cylindrical part graduated, *e.g.*, from the 6 to the 8 lb. graduations, thus providing a more open scale. If gases of quite different heating values are to be tested, several such graduates of different volumes might be desirable, or it may be that weighing the water would prove more convenient under such circumstances.

Standard specifications for glass volumetric apparatus may be found in Bureau of Standards *Circular* No. **9**. Glassware is usually graduated in cubic centimeters at some standard temperature. Graduates subdivided in pounds and decimal fractions of a pound and suitable for use with calorimeters would, so far as is known to the Bureau, have to be made to order.

4. *Graduate for Measuring Condensate.*—A 25-c.c. glass graduate of the usual form is suitable for this purpose. Graduates conforming to the Bureau specifications (*Circular* No. **9**) are to be preferred.

5. *Gas Meter.*—The points to be considered in the choice of a laboratory gas meter are considered on page 532.

6. *Pressure Regulators.*—Two types of wet pressure regulators are furnished with flow calorimeters: the simple float type, consisting of a small gas holder, the pressure of the gas on the inside of the holder actuating the

gas valve connected therewith; and the counterbalanced float type, in which the gas holder, suspended from one arm of a balance, is counterbalanced and may be compensated so as to maintain a constant pressure for all positions. In the latter type the gas pressure desired on the outlet side of the regulator is attained by varying the counterbalancing weights, and in the former type this is done by weighting the gas holder with suitable weights. In both types the pressure which is controlled by the regulator is the outlet pressure, which is the pressure of the gas within the holder. The simple float type, when properly constructed, is quite satisfactory.

7. *U-Gage.*—A U-gage on which the pressure in inches of water at the meter inlet may be read should be provided. Some of the float pressure regulators have a U-gage mounted so as to indicate the outlet pressure, and if one of these regulators is connected between the gas supply and the meter, the separate U-gage will be unnecessary. Some meters are furnished with U-gages which indicate the pressure at the meter outlet. While the pressure under which the gas is metered is the pressure at the meter inlet, no appreciable error would ordinarily be introduced in a calorimetric measurement due to the use of a gage indicating the pressure at the meter outlet.

8. *Barometer.*—Satisfactory mercurial barometers will be found listed in the catalogues of a number of thermometer and instrument makers. A barometer that can be read to 0.02 in., or even 0.05 in., will suffice for most industrial testing. The bore of the barometer tube, in the region where the position of the meniscus is read, should be not less than three-eighths of an inch. The barometer should be provided with an attached thermometer.

A barometer should be carefully tested before it is put into use, since the scale may be in error, the setting of the fixed point defining the position of the lower mercury surface may not correspond exactly to the zero reading of the scale, etc. An error of 0.05 in. in the barometer reading will cause an error of 1 part in 600 in the estimated volume of gas burned in a test—*i.e.*, 1 B.t.u. in a 600 B.t.u. gas. If the user can not arrange to have his barometer tested, he should at least make a series of comparisons with the barometer readings of a near-by station of the Weather Bureau, reading his own barometer at the same time of day and making any allowance that may be required for difference in level of his station and that of the Weather Bureau, and correcting his barometer readings to inches of mercury at 32°F.

It is always advisable, except possibly in traveling inspection work, to use a mercury barometer instead of an aneroid barometer, although the latter may be used if suitable precautions are observed. The user of an aneroid barometer is apt to be deceived by the fact that the scale of the instrument is readable to 0.01 in. or even closer. Its indications, however, will depend not alone on the pressure at the time it is read, but also on the pressure and the rate of variation of the pressure for some time previous, on how well it is compensated for temperature changes, whether it has been jarred sufficiently to derange its working parts, etc. With the average aneroid of high grade which has been carefully tested at a standardizing laboratory, and which has been again checked when received at its destination by a comparison with a standard barometer to insure that it has not been damaged in transportation, an accuracy of 0.05 in. may be attained in measuring the barometric pressure at a given station—*i.e.*,

under conditions where the aneroid is not subject to large and sudden variations in pressure as it is in altitude measurements. If an aneroid barometer is used, its readings should be frequently compared with the barometric pressure recorded at a nearby station of the Weather Bureau, due allowance being made for difference in altitude of the two stations. Further information relating to aneroid barometers may be found in Bureau of Standards *Circular* No. **46.**

9. *Psychrometer.*—Information relating to the various types of psychrometers and to their method of use may be found in *Circular* **48** of the Bureau of Standards. See also page 254.

10. *Gas Holder.*—If it is desired to test an average sample of gas collected over any period of time, a small gas holder must be installed, preferably in the calorimeter room. Such holders will be found listed in the catalogues of some makers of calorimeters and of gas appliances.

A holder of small capacity (about 2 cu. ft.) which is easily constructed may be used if a simple arrangement is provided by which the calorimeter may be brought to equilibrium conditions with gas drawn from the supply line, and then about 2 or 3 turns of the meter before the test is to be started the gas from the holder is turned on by a three-way stopcock. The 2 cu. ft. of gas will then suffice for four heating value tests. The gas in the holder, being in contact with water, may suffer a slight deterioration in its heating value if allowed to stand thus for a long time; the party making the tests should determine whether the effect of such deterioration is noticeable for the gas under the test, and make due allowance in interpreting results if the effect is of appreciable magnitude. (In this connection see *Tech. Paper* No. **36.**)

11. *Overhead Tank.*—The temperature of tap water is usually too variable and too different from room temperature to permit of its use as a source of water supply for flow calorimeters. A shallow overhead tank of at least 25 gal. capacity should therefore be provided as a source of water supply at room temperature. A copper or a copper-lined tank is preferable to an iron tank, in that it is more durable and is less likely to cause trouble by rust getting into the calorimeter. The tank should be shallow, both to promote uniformity of temperature throughout the water and to minimize variations in the rate of water flow. The tank should be covered to keep out foreign matter and a screen should be provided over the outlet which may well be an inch or so above the bottom of the tank, so as to keep the opening above any sediment that may deposit on the bottom of the tank. The tank should be provided with a suitable overflow connected to a drain and preferably with a water gage showing the depth of water. Unless the water supply is very clear it should be filtered into the tank. It will be found convenient to have a supply of both hot and cold water for filling the tank, and in this case there should also be provided, either some means for stirring the water in the tank or of running the hot and cold water in at the same time through a single inlet pipe in which a thermometer is mounted so that the tank can be filled with water at room temperature. In a laboratory where numerous heating value tests have to be made, a thermostat, such as is described in *Tech. Paper* No. **36,** for controlling the temperature of the water supply, is a convenience.

It is not necessary to install a water supply tank for the operation of the calorimeter. The calorimeter may be filled with tap water through special connections provided for that purpose. A small supply of cold water may be necessary for adjusting the initial temperatures of the calorimeters.

12. *Table.*—The calorimeter and its accessory apparatus may be mounted on a table of suitable size (*e.g.*, 30 by 60 in.), and of the usual height. While, perhaps, not strictly necessary, since the calorimeter should never be left in operation when the observer is absent from the testing room for any considerable length of time, it is an additional safeguard against fire to have the top of the table of stone or other fire-resisting material. The table should be provided with suitable plumbing connections to the overhead water supply to drain and to the supply of gas to be tested.

13. *Calorimeter Cabinet.*—If the calorimeter is of the flow type, it, together with the accessory apparatus, may be mounted in a special cabinet such as that described in the report of the committee on calorimetry, *Proceedings of the American Gas Institute*, **4** (1909), 205. The advantages of this form of mounting are that the apparatus is protected while not in use from dust and from fumes, if such be present, and that the cabinet may be locked. The cabinet contains an overhead tank which serves as a source of water supply to the calorimeter. Special calorimeter cabinets are also listed by some of the makers of calorimeters.

(*c*) **Portable Equipment.**—None of the calorimeters listed, with the exception of the Parr, are too cumbersome to be used as portable calorimeters in traveling inspection work. For traveling inspection work suitable carrying cases, such as may be obtained from the manufacturers of some of the calorimeters, should be used. It is better to have the gas meter, and if possible the calorimeter also, carried by hand, since the damage when carried by express may be serious, and it is impossible to transport a calorimeter as baggage with reasonable safety. A portable outfit, to be useful, would have to include practically the complete laboratory equipment mentioned above, with the exception of the gas holder, overhead tank, and table.

It is not absolutely essential that an inspector carry around with him a complete calorimetric equipment if one is to be found where the test is desired. A set of standardized thermometers, a standardized 1-lb. weight, or a standardized graduate, and a one-tenth cubic foot bottle, will afford the facilities necessary for checking the accuracy of the results found with any of the calorimeters listed. The barometer may be checked by comparison with the barometer at the nearest station of the Weather Bureau. An inspector familiar with the operation of flow calorimeters will readily detect any defect of the calorimeter.

STANDARDIZATION OF APPARATUS

(*a*) **Calorimeter and Accessory Apparatus.**—From the tests described in *Tech. Paper* No. **36,** it is seen that heating-value determinations with some types of calorimeters are subject to a constant error amounting to nearly 2 per cent. There can be no doubt, therefore, that it is desirable to have a calorimeter tested before it is put into use, although the behavior of the calorimeter in use will often give some indication as to its reliability. Im-

portant checks on the efficiency of a flow calorimeter may be obtained by observing whether the burner is set high enough, whether the combustion products are discharged at a temperature not too much in excess of inlet-water temperature, whether the normal rate of gas consumption is about what it should be for the type of calorimeter in question, and whether the outlet-water thermometer gives the true mean temperature of the effluent stream of water. This latter feature may be tested by moving the bulb of the thermometer so that it will occupy different positions in the cross-section of the effluent stream of water.

The calorimetric thermometers should be tested in a recognized standardizing laboratory, and the corrections so determined should be applied in computing the results of tests.

The graduate for measuring the condensed water should be tested; it should not be in error by as much as 0.2 c.c., so that the application of corrections to the volumes measured with it will be unnecessary.

The gas meter must be tested, and it is desirable, as stated elsewhere, that the user should occasionally recalibrate it, using one of the methods described on page 532. It may be possible to set the fixed mark to which the water level is adjusted so that the volume of gas delivered by the meter, as determined by a series of calibrations, will be equal to the nominal volume, within 0.2 or 0.3 per cent., in which case the application of the correction for error of the meter may be omitted. However, the accuracy of the meter in use is limited by the accuracy with which the water level can be readjusted to the fixed mark.

The barometer should be tested.

The thermometers of the psychrometer and of the barometer, and those used with the gas meter and for measuring the temperature of the products of combustion should also be tested, but it is possible to obtain thermometers which are sufficiently correct so that their errors may be neglected. If the thermometer in the gas meter is correct within 0.3°F., and if the other thermometers mentioned are correct within 0.5°F., no corrections need be applied to their readings.

The apparatus for weighing or measuring the water should also be tested. The weighing apparatus should be accurate to 0.1 per cent., so that the application of corrections will be unnecessary, but the application of the corrections may be necessary if volumetric apparatus is used.

(*b*) **Differential Correction for Time Changes of Calorimetric Thermometers.**—When thermometers for use with flow calorimeters are standardized, the corrections determined and certified are those applicable at the time of test. However, small changes in the volumes of the bulbs are likely to occur, making all corrections of a thermometer differ by a constant quantity from what they were at the time of the test. The manner of determining a differential correction, and thus eliminating from the calorimetric measurements the errors due to relative changes in the volumes of the bulbs of the thermometers, is shown in the following example:

Thermometer A has a certificate correction of −0.04° at 70°F. and thermometer B has a certificate correction of −0.06° at 70°F.

The two thermometers were held with bulbs adjacent in a well-stirred pail of water, a little below room temperature (70°), and the following

readings were taken. The thermometers were read alternately, at equal time intervals, beginning and ending with the same thermometer, in order that the means of the two sets of readings might correspond to the same instant of time:

	A	B
	69.43	69.48
	0.45	0.49
	0.46	0.50
	0.47	
Means	69.45	69.49
Certificate correction	− 0.04	− 0.06
Corrected readings	69.41	69.43

It will be seen that the corrected reading of B is 0.02° higher than that of A. A differential correction of −0.02° must therefore be applied, in addition to the certificate corrections, to all readings of thermometer B. The differential correction should be calculated for and applied to the readings of the outlet-water thermometer, thus:

	A	B
Mean of thermometer readings	69.45	69.49
Certificate correction	− 0.04	− 0.06
Differential correction		− 0.02
Corrected temperatures	69.41	69.41

The differential correction should be determined about once a month. It should be determined at, or very near, some temperature at which the thermometers have been tested, in order to avoid introducing into the differential correction any errors resulting from interpolation between two certificate corrections.

(*c*) **Determination of Normal Rate of Gas Consumption.**—The normal rate of gas consumption for the kind of gas to be tested and for the calorimeter in question, operated with the damper set as it is to be subsequently used in testing the gas, should be determined before the calorimeter is put into service and should be redetermined from time to time thereafter (at intervals of a few months is sufficient). The normal rate has been defined as 70 per cent. of the rate at which combustion begins to be incomplete. (See *Tech. Paper* **36.**)

A convenient procedure for determining the rate at which combustion is no longer complete is as follows: With the calorimeter in operation the rate of gas consumption is increased step by step—*e.g.*, by successively increasing the number of weights on the pressure regulator. As long as combustion is complete, the effect of increasing the gas rate is to cause an increment in the reading of the outlet-water thermometer. However, when the gas rate attains such a value that combustion is no longer complete, there will be a decrement in the reading of the outlet-water thermometer accompanying an increase in the gas rate. The gas rate at which combustion begins to be incomplete (for a given damper setting, etc.) ca

thus be determined to within a few per cent. An almost equally good test is furnished by the odor of the combustion products, as the odor due to incomplete combustion is readily perceived for most gases.

SETTING UP APPARATUS

If the calorimeter is of the usual flow type, it should be set up about midway of the length and forward of the center line of the table, in such a position as to leave room at the left for the pressure regulator and the meter, and on the right for the weighing bucket and scale, or for the graduate, if the latter is used instead of the scale.

(*a*) **Water Connections.**—Temporary connections from the overhead tank to the inlet of the calorimeter and the necessary connections to drain may be made by means of rubber tubing. In a permanent laboratory installation it is preferable to use piping, making the connections between the ends of the pipes and the calorimeter with short lengths of rubber tubing. Ordinarily $\frac{3}{8}$-in. pipe with suitable nipples for rubber tube connections will be found satisfactory for the various water connections. If the calorimeter has an inlet overflow weir, it is some convenience to have a short length of glass tubing in the line leading from this weir to drain. This affords a ready means of determining whether sufficient water is being delivered to insure that no air will be carried down into the calorimeter with the water and thus cause variations in the water flow. All valves and cocks in the supply pipes to the overhead tank and from the overhead tank to the inlet of the calorimeter should be so located as to be conveniently accessible to the observer, so that the necessary manipulations can be made without endangering the thermometers or other parts of the apparatus.

(*b*) **Gas Connections.**—The pressure regulator may be connected either between the gas supply and the meter or between the meter and the burner. The former location has the following advantages: (*a*) The pressure at meter inlet is constant; (*b*) leak tests are more easily made; (*c*) the effect of leaks in the regulator or its connections are eliminated; (*d*) the effect of variation in volume of the regulator, due to motion of the drum, is eliminated; (*e*) the U-gage on the regulator may be used to measure the gas pressure at the meter inlet. The principal reason for putting the regulator between the meter and the burner is to eliminate the effect of fluctuations in gas pressure due to the operation of the meter. With a well-constructed meter in good working condition the fluctuations which it introduces are scarcely noticeable in their effect on the readings of the outlet-water thermometer.

If a wet-pressure regulator is connected between the meter and the burner, a similar regulator should also be connected between the gas supply and the meter, unless the gas supply is drawn from a gas holder in the laboratory. It is desirable for a number of reasons that the pressure of the gas in the meter should not exceed 2 or 3 in. of water. It has been found that a wet-pressure regulator of proper construction may be used to reduce the pressure from 10 or 12 in. of water to the pressure required at the burner. Under any circumstances the gas should not be shut off beyond the regulator, if the pressure is sufficient to break the water seal.

The gas meter should be set up so as to be viewed normally by the observer when manipulating the change-over device; *i.e.*, the meter dial will

not, in general, be parallel to the front edge of the table. A well-illuminated white background should be provided back of the gage glass or the sight box. It is not well, however, to attempt to secure this background by the use of a milk-glass background in the sight box itself, since under such circumstances the adjustment of water level is difficult.

Connections should be made to a U-gage to indicate the pressure, in inches of water, at the meter inlet. As shown in *Tech. Paper* **36,** the pressure at the meter inlet may be so adjusted that the total pressure of the gas in the meter is equal to the observed barometer reading; this is done by making the excess of gas pressure above atmospheric pressure equivalent (*i.e.*, equal and of opposite sign) to the temperature correction to the barometer reading. Even if this method of compensating the barometer corrections is not used, the observations and computations will be somewhat simpler if the pressure of the gas at the meter inlet is kept constant.

The necessary connections between the gas supply, the service or other pressure regulator, the meter, and the wet-pressure regulator may be made with ordinary ¼-in. pipe, or suitable metal tubing, the ends of the pipe or tubing being connected to the apparatus by short lengths of rubber tubing or by suitable unions. However, connections that are too rigid and difficult to disconnect may prove a source of inconvenience. The connection to the burner must be flexible to permit the withdrawal of the burner from the calorimeter. Thick-walled rubber tubing may be used throughout, provided care is taken to saturate the tubing with the gas before making tests. The use of metal hose has been recommended for the connections, and it is undoubtedly desirable from the point of view of reducing the fire hazard, but such tubing is very often found to leak slowly and should therefore be carefully tested for leaks before use. Small leaks in the connections ahead of the meter, of course, do not affect the results of heating-value determinations.

DIRECTIONS FOR OPERATING FLOW CALORIMETERS

(*a*) **Manipulation and Observations.**—It is assumed that provision has been made for obtaining a representative gas supply. If an average sample of the gas is to be tested, this must be collected in a small holder, the supply being drawn from a line from which gas is being used, to insure that fresh gas is collected, at a rate which will give a sample of not less than 2, and preferably as much as 5, cubic feet for the test; but if gas for the test is drawn direct from the supply line, it is only necessary that the connections be flushed free from old gas as directed below, so that the test will be made on gas representative of that in the mains. Having the gas supply thus properly provided and the calorimeter set up as directed in the previous section, each test is made in the following manner:

1. *Free gas connections from old gas* by use of "blow-off" if this is provided, or by starting several burners to rapidly burn out the old gas which is in the supply pipe. When burning the gas for this purpose, it is well to use an appliance connected to the same supply line, but located in an adjoining room, in order to avoid large temperature changes in the room where the test is to be made. It is well to *burn one burner continuously throughout*

the period of test, connecting this burner to an outlet near the point from which the gas supply for the calorimeter is drawn.

2. *Observe temperature of laboratory* in the vicinity of the calorimeter, using a thermometer which is shielded from radiation or drafts from hot or cold objects near by, such as cold walls, windows, radiators, or even the heated body of the calorimeter itself. The reading of the dry bulb thermometer of the psychrometer, used as in the determination of the atmospheric humidity, can be taken as the room temperature. The observation taken at this time is not entered on the record, but is used merely to enable the observer to adjust the temperature of the gas meter and of the water supply.

3. *Adjust the gas meter for: Temperature; level; position of index relative to drum; water level; and saturation of water with gas.* The meter temperature, as indicated by a thermometer, the bulb of which is in the water in the meter, should not differ from room temperature by more than 3°F.; it may be adjusted by pouring in warm or cold water and then running gas through the meter for a few minutes to thoroughly stir the water. The screw plug at the bottom of the "dry well" at the back of the meter should be removed occasionally to drain off any water accumulated there. The meter is leveled as judged by levels of plumb bob attached; and this leveling must be carefully done in the same manner as the leveling at the time of calibration of the meter. Frequently, preferably before each test, it should be determined that the index is in the proper position relative to the drum of the meter, and adjustment should be made, if necessary. The level of the water in the meter must be adjusted as accurately as possible *before each test*, unless several tests are made on the same day, the adjustment being made in the same manner as is done before calibration of the meter. When verifying adjustment of water level the index should be in the zero position and the inlet and outlet of the meter *must be open to the air*. When a meter is filled with fresh water, the absorption of gas by the water usually causes no significant effect on the heating value after 2 or 3 cu. ft. of gas have been passed through the meter. If a meter has been shipped or carried from one laboratory to another, it should be recalibrated before use in determinations of heating values. It will probably be found inconvenient to recalibrate the meter each time it is used, although a somewhat higher accuracy may be attained by following this procedure.

4. *Test for gas leaks.* If the wet-pressure regulator is connected between the gas supply and the meter, this test may be made as follows: Remove the burner from the calorimeter and, after passing from 0.1 to 0.2 cu. ft. of gas through the meter to insure that any explosive mixture of gas and air has been removed from the meter, light the gas. The movable drum of the pressure regulator should be displaced up and down several times to remove air and old gas contained in the regulator. After the meter has made three or four revolutions, shut off the gas at the base of the burner and immediately afterwards shut off the gas supply to the regulator to prevent the gas pressure from breaking the water seal of the regulator. The regulator will contain a sufficient volume of gas for the leak test. If during an interval of 10 minutes the meter index does not show a movement exceeding one one-hundredth of a revolution (0.001 cu. ft. leakage) the leakage may be considered negligible. While waiting for this test the adjust-

ments required in paragraphs 5 and 6 may be made. The test for leaks should be made each day. It is evident that only leaks in the meter or between the meter and the burner (base of flame) affect the determination of heating values, and only such leaks are detected by the above test.

If the wet-pressure regulator is connected between the meter and the burner, a constant gas pressure, which is not high enough to break the water seal of the regulator, must be provided at the meter inlet in order that the test for leaks may be made. Such a constant pressure may be obtained in the manner described above from another wet-pressure regulator connected between the gas supply and the meter or from a gas holder in the laboratory. It is necessary to depress the float of the regulator connected between the meter and the burner to allow the pressures on both sides of the valve of this regulator to equalize before taking the initial meter reading for the leak test, as otherwise there probably will be a slow creeping of the meter index due to leakage through the valve of the regulator.

5. *Adjust temperature of water in supply tank.* The water supplied to the calorimeter should be within 2°F. of the laboratory temperature, and if the water in the tank is not within this limit it should be adjusted by adding hot or cold water, as may be required, and stirred to insure uniformity of temperature throughout the tank. When the tank is filled after one day's test is completed, the water will come to room temperature for the next day's test if the room temperature is quite uniform in the interval; but in any case where hot and cold water are available, the adjustment to the proper temperature can be made easily. If the water temperature is 5°F. different from the laboratory temperature, it will make a difference of about 3 B.t.u. in the observed heating value. In this connection see also page 526.

6. *Start flow of water through calorimeter and expel air from water circulation system.* With the inlet-water valve of the calorimeter set at approximately the position for regular operation, turn on enough water so that some will be discharged to the drain from the overflow weir at the inlet of the calorimeter. To promote the escape of air from the water-circulation system, open and close the inlet-water valve on the calorimeter a number of times, and if necessary in order to remove air from about the thermometer bulbs lift the stoppers holding them in place, so that all the air and a small amount of water will be forced out. If the calorimeter has an inlet weir, the water supply must be sufficient to provide for a continuous overflow from this weir during the above manipulation in order to avoid drawing more air into the calorimeter. Air entrapped in the water-circulation system is apt to cause fluctuation in the rate of water flow, with resulting fluctuations in the outlet-water temperature. Occasionally before the burner is inserted—operation 7—the water should be run through the calorimeter for a considerable time to determine whether there are any leaks in the water-circulation system.

7. *Light gas, adjust rate of gas consumption, adjust air mixer on burner, and insert burner into calorimeter.* When it is determined that no leakage is occurring, as described in paragraph 4 above, the burner is lighted and the flow of gas is adjusted to the *normal rate* (within ± 5 per cent. is sufficient) previously determined for the kind of gas and the calorimeter used and for the particular damper setting, etc., with which the calorimeter is operated.

The air supply to the burner is adjusted so that the luminous tip of the flame just disappears, and the burner is inserted in the calorimeter, being placed at the proper height on its support.

8. *Adjust water flow and set damper.* The rate of water flow is adjusted so that the rise in temperature of the water will be 15°–20°F., and the damper is set in the position determined in fixing the normal rate for this calorimeter. A lowering of 2 per cent., or even more, in the observed heating value may result from leaving the damper open when the rate of gas burning is that determined for a closed perforated damper. The magnitude of this lowering would depend not only on the construction of the calorimeter, but also on the character of the gas and the humidity of the air of the laboratory.

9. *Allow time for establishment of a condition of thermal equilibrium.* The time required for the attainment of thermal equilibrium is about 5 minutes for calorimeters of the Junkers, Hinman-Junkers, or Sargent type, somewhat longer for the English type of Simmance-Abady calorimeter, and from 20 to 30 minutes for the Boys and the American type of Simmance-Abady calorimeter. If net heating values are to be determined, it will be necessary to wait until the condensed water from the products of combustion has begun to drain regularly from the calorimeter. During this interval most of the observations and records called for in the next paragraph may be made.

10. *Prepare record sheet and make the preliminary observations.* The record form, which is illustrated on page 524, is prepared for use in the test by entering the following items:

Date, time, place of test, and kind of gas;

Identification marking (number or name) of calorimeter, meter, and thermometers;

Statement whether the gas line has been purged and whether proper tests have been made for leaks and for meter adjustment; and

Position of water valve[1] and of damper (by number of scale division, or by "open" or "closed" in case of damper).

The "preliminary observations," as they may be called, should be made during the few minutes just preceding the period when the condensed water is collected; these observations include the following made in the order listed:

(*a*) Temperature of the barometer (to the nearest 1°F.).

(*b*) Barometric height (to nearest 0.01 in.).

(*c*) Gas pressure at meter inlet (to nearest 0.1 in. of water column).

(*d*) Reading of meter thermometer (to the nearest 0.1° or 0.2°F.).

(*e*) Readings of the wet and dry bulb thermometers of the psychrometer, as directed on page 254.

(*f*) Reading of products thermometer (to the nearest 1°F.).

(*g*) Time in seconds of one revolution of the index of the meter.

11. *Begin collection of condensate.* As the index of the meter passes through the zero position, place the 25-c.c. graduate under the drain tube of the calorimeter and record the meter reading (*i.e.*, total revolutions).

[1] This is recorded largely as a matter of convenience for the operator. Examination of the records extending over a considerable period might show whether the water passages were becoming clogged.

12. *Take the first series of observations*, which is carried out in the following manner:

(*a*) Record one or two readings each of inlet and outlet water thermometers before the meter index has completed the first revolution after the collection of condensate is begun.

(*b*) As the meter index passes through the zero position, shift the change-over device to divert the effluent stream of water from drain to measuring vessel. The operations of shifting the change-over device, both on and off, should be made in the same manner and with the greatest care, so that the time interval between the two operations shall be as nearly as possible equal to that defined by the passages of the meter index past the zero position of the dial.

(*c*) Read the inlet and outlet water thermometers alternately at approximately equal intervals of time during the two revolutions of the meter index. If the thermometer readings are spaced so that the interval between them corresponds to the time required for the index of the meter to pass over about 10 divisions of a dial having 100 divisions, a sufficient number of readings will be obtained. If the inlet-water temperature is fairly constant, this procedure will give more readings of the inlet-water thermometer than are really required, in which case one reading of the inlet-water thermometer at the beginning, a second reading at the completion of the first revolution of the meter index, and a third reading on the completion of the second revolution of the meter index may be taken instead of alternate readings as above indicated. If the variations in inlet-water temperature are sufficiently large so that the average of the three readings does not give a sufficiently accurate average of the inlet-water temperature, it may be found that the average of the alternate readings may not give results of the desired accuracy, in which case the method of reading used by Immenkötter (see page 517) must be adopted.

(*d*) At the instant the index of the meter passes through the zero position, on the second turn after the water was turned into the measuring vessel, shift the change-over device diverting water from measuring vessel back to drain.

(*e*) Record one or two readings of the outlet-water thermometer.

(*f*) Weigh the water collected and record the weight (to the nearest 0.01 lb.); or observe and record its volume (to the nearest 0.01 lb. or nearest 5 c.c.). If a cylindrical graduate is used, great care in reading will be required to keep the error within the above limits. If a special graduate with enlarged bottom and open scale is used, the required accuracy may be readily attained.

13. *Take the second (and later) series of observations.* As soon as convenient after the first series is complete repeat the full set of observations of operation 12. At least two series of observations should be made for a test.

14. *Stop collection of condensate* by removing the small graduate from under the drip tube of the calorimeter just at the instant the index of the meter passes the zero position after not less than 7 (or preferably 10) revolutions since beginning the collection of the condensate. Record meter reading (total revolutions) and the amount of water collected (to nearest

0.1 or 0.2 c.c.). If the net heating value is to be determined with the same accuracy as the total heating value, another collection of condensate similar to the first should be made, in order that the result may not depend upon a single observation.

15. *Repeat the preliminary observations of operation* 10. This is done on the general principle that no single observation should be accepted without some check, and, secondly, because it enables the observer to decide whether the determination should be repeated on account either of a mistake in the first readings or on account of a change in conditions sufficient to affect the results.

16. *Turn off gas and then turn off water.* The apparatus may be stopped at once after completing a test; or if the water supply in the tank is ample, the calorimeter may be left running until the calculation of the test data has been completed so that a repetition of the test may be made at once in case there is some reason for desiring to verify the whole test. In any event, it is well before shutting down the apparatus to glance over the test record sheet and be sure that no portion of the record is incomplete.

Caution.—In operating a flow calorimeter great care must always be taken, and especially when starting or stopping a test, to insure that the gas is not burning in the calorimeter when the water is shut off.

Notes on reading the thermometers. It is preferable to make an estimate of the average reading of the outlet-water thermometer during each interval, and to record this average as the reading rather than to record the readings observed at certain instants. The inlet and outlet water thermometers should be provided with suitable reading glasses and the observer should estimate the thermometer readings to one-tenth of the smallest graduation interval; *i.e.*, if the thermometer is graduated to 0.1°, the readings should be made to 0.01°. With a little practice this can be so easily done that it is to be preferred to reading the nearest one-half graduation interval. To avoid error due to parallax, the line of sight must be perpendicular to the thermometer stem, a condition which will be secured with a properly constructed reading glass, if the mercury meniscus of the thermometer is in the center of the field of view.

To minimize the fluctuations in the readings of the outlet-water thermometer, due to drafts and to air currents, the calorimeter should be shielded from drafts and the observer should move about as little as possible while taking the thermometer readings.

An error of 0.1° in the determination of the average temperature of either the inlet or the outlet water or a combined error of 0.1° in the two temperatures will cause an error of one part in 150, *i.e.*, 4 B.t.u. with a 600 B.t.u. gas, if the temperature rise is 15°.

Operation of Flow Calorimeters if Inlet-water Temperature is Variable.—It is always preferable to keep the variation of temperature of the inlet water during a single complete series of observations within 0.05°F., which can easily be done if care is taken to have the water in the overhead tank at a uniform temperature throughout when the test is started.

Under the usual methods of operation, where inlet and outlet water temperatures are read alternately, the errors introduced into heating-value

tests by variations in the inlet-water temperature will depend not alone on the magnitude of such variations but also on the water content of and the rate of water flow through the calorimeter that is used in the test.

The effect, on the observed heating value, of variations in the inlet-water temperature may be very materially reduced, even if not wholly eliminated, by the procedure recommended by Immenkötter, *viz.*: Each of the readings of the inlet-water thermometer should be made earlier than indicated in the directions already given by an interval of time equal to that required for the water to flow through the calorimeter, this interval being equal to the water content of the calorimeter divided by the rate of flow of water.

The method recommended by Immenkötter is a desirable one under any conditions of operation. If the temperature of the inlet water is variable and also differs considerably from the room temperature, it will be necessary, besides reading the thermometers as above indicated, to make additional corrections as described in the following section.

Operation of Flow Calorimeters if Inlet-water Temperature Differs Considerably from Room Temperature.—In a permanent laboratory installation, such as that outlined herein, there should be no difficulty in adjusting the temperature of the inlet water so that it shall be nearly equal to room temperature, thus making very small the error introduced into the results of heating-value tests due to the lack of equality of the two temperatures. Under some conditions, as in traveling inspection work, it may be necessary to make tests under less favorable conditions, in which case fairly good results could still be obtained by applying suitable corrections in calculating the total and the net heating values, to allow for the effect of a considerable difference between inlet-water temperature and room temperature. Even when an overhead tank is used to supply the water to the calorimeter there will often be a difference of a few degrees between the temperature of the water and the temperature of the room. A considerable increase in accuracy may be obtained by making a correction for the heat interchange due to this difference, and it is much easier to apply the correction than to secure exact agreement between inlet-water and room temperatures.

(*b*) **Records and Computations.**—The observed data and the important steps in the computations of a heating value test, made up of two or three series of temperature observations, as described above, may be entered in a record form similar to that shown on page 524. This form may conveniently be on an 8 by 10 in. sheet, or, if it is desired to file the records in a way corresponding to the other test records, on a 5 by 8 in. card, utilizing both sides of the card.

For official testing it is essential that the original record, containing all the observed data, should be kept. Numerous forms of abbreviated record cards have been used which either do not contain all the essential data, or which require the transfer of the computed data to the record. In either case the result entered on such record can not be as conveniently checked back to the original observations.

The computations will be very much facilitated if all the calibration data of calorimeter, meter, thermometers, barometer, etc., are entered

on a card for convenient reference. If this is done, the application of the corrections is so simple that there is no justification for omitting them. The original certificates of test should be preserved for reference in a safe place and should not be used in making routine computations. It is also desirable to have conveniently accessible the necessary tables and charts. A complete set of tables for this work would be made up of the following, some of which are given in this treatise.

Corrections for reduction of barometric heights to standard temperature (page 530).

Reduction of pressures in inches of water to equivalent pressures in inches of mercury.

Correction factors for gas volume (page 527).

Relative humidities for various dry and wet bulb readings of ventilated psychrometer (page 502).

Rate in cubic feet per hour corresponding to various times (in seconds) of revolution of the meter index.

Emergent stem corrections to readings of outlet-water thermometers (page 526).

Corrections (in B.t.u.) to be applied to observed heating values in calculating total heating values of illuminating gases (about 600 B.t.u.) for various atmospheric humidities and temperatures (page 529).

The card of calibration data referred to in the calculation would contain a number of small tables and memoranda, thus assembling in convenient form the following information:

Calorimeter number, meter number, thermometer numbers, position of water valve under ordinary condition, damper position which corresponds to that used in determining normal rate, the date of the determination of the differential thermometer correction and the correction itself, date of last meter calibration, table of certificate corrections for barometer, certificate corrections for inlet and outlet water thermometers, the correction for heat loss from calorimeter, and such other data as the inspector found was needed from day to day in routine computation.

The calculation may be performed as follows, although the order of the operations may, of course, be varied. When the computer becomes familiar with the reasons for the various steps in the calculations it may be preferable to follow a different sequence; *e.g.*, it would probably be more convenient to enter on the records at one time all the data to be taken from the card of calibration data, such as constant correction to barometer reading, correction to meter thermometer, certificate correction to inlet and outlet water thermometers, the differential correction to outlet-water thermometer, the meter calibration constant, and correction for heat loss from calorimeter. The sequence given below was adopted because it admits of a more logical presentation in explaining the reasons for the various operations.

1. Enter from the card of calibration data the *certificate correction* and, from page 530, the *temperature correction* to the barometer. The corrections thus entered are added to (or subtracted from) the *barometer readings* to find the *corrected barometric height*, the *average* obtained from the two barometer readings being entered on the record.

The certificate correction is that determined in a test of the barometer, and in a well-constructed instrument this correction should not exceed 0.02 in., in which case the correction may be omitted. The temperature correction must be applied to the observed reading of a mercury barometer on account of the thermal expansion of the mercury and of the brass scale.

2. Enter the *equivalent, in inches of mercury*, of the pressure at meter inlet, and add this to the *corrected barometric height* to find the *total gas pressure.*

The figures in the above calculations should be carried out to 0.01 in. of mercury.

The table for reducing the pressure, in inches of water, at the meter inlet, as read from the U-gage, to the equivalent in inches of mercury, is calculated by dividing the pressure expressed in inches of water by 13.6 (the density of mercury). This correction for gas pressure in the meter and the temperature correction for the barometer may both be omitted if they are made equal and of opposite sign.

3. Enter from the card of calibration data the *certificate correction* to meter thermometer; and calculate the corrected *meter temperature*, only the average calculated from the two readings of the meter thermometer being entered on the record.

4. From page 527 enter the *reduction factor, F* (to the third decimal place), corresponding to the *total gas pressure* and the *meter temperature.*

The gas is measured at a temperature entered in the record as the meter temperature and at a pressure entered in the record as the total pressure of the gas, the volume being equal to the number of revolutions of the meter index (*viz.*, 2) multiplied by the calibration constant of the meter (entered on the record as 1 revolution of meter = . . .). Multiplication of this measured volume by the factor F gives the volume which the same quantity of gas would have occupied at a temperature of 60°F. and under a total pressure of 30 in. of mercury.

5. From page 502 enter on the record the relative atmospheric *humidity* (to the nearest 5 per cent.) corresponding to the recorded *dry bulb* and *wet bulb* thermometer readings of the ventilated *psychrometer.*

The thermometers of the psychrometer should not be in error by as much as 1°F., and if such is the case no certificate corrections need be applied to the observed readings. The reading of the dry bulb thermometer should be taken as the room temperature whenever that temperature is used in the subsequent calculations.

6. The temperature of the products of combustion is ordinarily not used in the calculation, but is of value in showing whether or not the calorimeter was operating as it should. In the better types of flow calorimeters the excess of temperature of the products above the temperature of the inlet water is less than 2°F. if the burner is properly placed in the calorimeter. Even when the excess of temperature is 4° to 5°F., the resulting error, due to neglecting this heat loss, as is done in these calculations, is only a few tenths of 1 per cent. of the total heating value.

7. Enter the *rate* in *cubic feet per hour* corresponding to the *time* in seconds of *one revolution* of the meter index.

The rate of gas consumption in cubic feet per hour may be calculated as follows:

$$\text{Rate (for a } \tfrac{1}{10}\text{ cubic foot meter)} = \frac{360}{\text{time of 1 rev. in secs.}}$$

$$\text{Rate (for a } \tfrac{1}{2}\text{ cubic foot meter)} = \frac{300}{\text{time of 1 rev. in secs.}}$$

The rate is entered on the record to show that the calorimeter was operated at about its normal rate (within ±5 per cent.). The calibration correction of the meter need not be taken into account in the calculation of the rate, unless, of course, that correction is unusually large.

8. Average separately, for each series, the *readings of the inlet and of the outlet water thermometers*, omitting the *preliminary* and *supplementary* readings, and enter these *averages* on the record.

9. Enter from the card of calibration data the *certificate corrections* corresponding to the average readings of the inlet and outlet water thermometers.

10. Enter from the card of calibration data the *differential correction* to the reading of the outlet-water thermometer.

This correction, determined as described on page 508, is applied in order to correct for any relative change that may have occurred in the thermometers since the determination of their certificate corrections.

11. Enter on the record from page 525 the *emergent stem correction* to the average readings of the outlet-water thermometer.

Correction for Emergent Stem of Thermometer.—If the mercury thread of a thermometer is not at the same temperature as the bulb, the emergent stem correction may be too large to be neglected. The correction for emergent stem may be computed from the formula:

Stem correction $= K \times n(T° - t°)$.

K = factor for relative expansion of mercury in glass; 0.00015 to 0.00016 for Centigrade thermometers, 0.000083 to 0.000089 for Fahrenheit thermometers, at ordinary temperatures, depending upon the glass of which the stem is made. For ordinary calorimetric thermometers graduated on the Fahrenheit scale, the value $K = 0.000085$ may be used.

n = number of degrees emergent from the bath.

T = temperature of the water.

t = mean temperature of the emergent stem.

If a flow calorimeter is operated with the inlet water at room temperature, the stem correction for the inlet-water thermometer will obviously be zero, while the outlet-water thermometer will read too low. Under the conditions stated, the stem correction to be applied to the readings of the outlet-water thermometer will depend upon the temperature of the room, the temperature rise, and the point on the scale to which the thermometer is immersed.

The stem corrections to be applied to the readings of the outlet-water thermometer are given on page 525. In computing the table some allowance was made for the fact that the mean temperature of the stem of the outlet-water thermometer is slightly above that of the room.

It is evident that, if the inlet water is not at room temperature, the readings of both the inlet and outlet water thermometers will be subject to stem corrections. If the two thermometers are both immersed to the same graduation, and, if the inlet water does not differ by more than 5°F. from the room temperature, the application of the tabular corrections, page 525, to the readings of the outlet-water thermometer will lead to the same results for the temperature rise as applying the two stem corrections independently. If, on the other hand, the two thermometers have each the same number of degrees emergent, the application of the tabular corrections to the readings of the outlet-water thermometer will lead to the same result as applying the two stem corrections independently, regardless of the difference between inlet-water temperature and room temperature. The statements in this paragraph may be readily verified by actually making the computations.

In the example cited on the record sheet, page 524, the stem corrections may be computed by the formula given above, or they may be taken from the table. The temperature of the inlet water ($T = 67.93$) being practically equal to room temperature, ($t = 68$) $T° - t°$, is very small and hence the stem correction to the inlet-water thermometer is negligible. The outlet-water thermometer used in the test was immersed to the 30° mark so that, n, the number of degrees emergent, was 86° − 30° = 56°. The bulb temperature was 86° −

68° = 18° above that of the room; and since the mean temperature of the emergent stem was a little above that of the room, on account of heat received from the calorimeter, $T° - t°$ was equal to 17°, and hence the emergent stem correction to the outlet-water thermometer = 0.000085 × 56 × 17 = +0.08°F. The correction interpolated from page 525 is also +0.08°F.

12. Enter on the record the *corrected temperature* of the inlet and of the outlet water for each series by applying the several corrections to the averages of the readings of the inlet and outlet water thermometers.

13. Subtract the corrected temperature of the inlet water from the corrected temperature of the outlet water for each series, and enter this *temperature rise*, T, on the record.

14. The weight of *water collected*, W, is corrected if calibration has shown any error in the weights used.

Such correction, however, should not be necessary. In general, the correction for buoyancy and the correction for the variation of the specific heat of water may be neglected, since both corrections are small and are, for the conditions under which flow calorimeters are used, of opposite sign.

15. Opposite *number of revolutions of meter* enter on the record the number of revolutions made by the meter index during the time the effluent water for each series was being collected.

16. Enter from the card of calibration data the meter calibration constant, *viz.*, the number of *cubic feet corresponding to one revolution* of the meter.

17. Enter on the record the *gas volume*, V, which is the product of the number of revolutions of the meter and the meter calibration constant.

18. Calculate and enter on the record for each series the observed heating value found from the following equation:

$$\text{Observed heating value} = \frac{\text{Water heated } (W) \times \text{Temp. rise } (T)}{\text{Gas volume } (V) \times \text{Reduction factor } (F)}.$$

Heating-value Computer.—The arithmetical work involved in the computations may be greatly diminished by the use of the heating value computer, which is illustrated in the *Proceedings of the American Gas Institute*, **3** (1908), 373. The computer is a circular slide rule with which the observed heating value may be read directly when the temperature and the total pressure of the gas, the volume of gas indicated by the meter, the weight of water heated, and the rise of temperature of the water are known. The computer can be read with an accuracy of 1 or 2 B.t.u., and if a few check computations are made occasionally no significant error should be introduced due to the use of the computer. The computer is published by the United Gas Improvement Co., Broad and Arch Streets, Philadelphia, Pa.

Agreement of Duplicates.—In general, 2 or 3 series of observations should give observed heating values which do not differ from one another by more than 6 B.t.u. for a 600 B.t.u. gas. Greater variation is sometimes allowable if it is impossible to get good conditions for work, as, for example, if a water supply of uniform temperature is not available. But, in general, results which do not agree well within 6 B.t.u. indicate either that the operator has neglected some important precaution or that the apparatus is not in good condition.

19. Enter from the card of calibration data the *correction for heat loss.*

The heat lost from the surface of a calorimeter, due to the average excess of the surface temperature above that of its surroundings, is often called the radiation loss. The radiation loss, when the inlet water is at room temperature and the temperature rise is 15° to 20°F., is of the order of 0.1 or 0.2 per cent. (*i.e.*, about 1 B.t.u. for a 600 B.t.u. gas) of the total heating value of the gas for the Junkers (original and new types), Hinman-Junkers, Sargent, and Simmance-Abady (English type) calorimeters, and may therefore be neglected or applied as desired. For the Boys and the Simmance-Abady (American type) calorimeters a correction for heat loss should be applied. (See *Tech. Paper* **36**.)

20. Enter from page 529 the *correction for effect of atmospheric humidity*, using the correction corresponding to the room temperature (reading of dry bulb thermometer of psychrometer) and to the percentage *humidity* as entered on the record.

If natural gas (about 1000 B.t.u.) is being tested instead of illuminating gas (about 600 B.t.u.), the correction for effect of atmospheric humidity should be taken from page 529.

The correction for the effect of atmospheric humidity is fully considered in *Tech. Paper* **36.** The necessity for a correction for the effect of atmospheric humidity is evident from the following considerations: On a dry day the air entering the calorimeter will carry in very little water vapor, while the products of combustion, being practically saturated at the temperature at which they escape from the calorimeter, will carry off a considerable amount of the water vapor formed in combustion, and the latent heat of condensation of this water vapor will not be measured in the calorimeter. On a day of high humidity the amount of latent heat thus lost will evidently be less, and under conditions of very high humidity more water vapor will be carried in by the air and gas than is carried out by the products, and heat will thus be imparted to the calorimeter.

The tabular values apply for the condition that room temperature, inlet-water temperature, gas temperature, and products temperature are all equal. In using the table, the correction corresponding to room temperature is taken; since if the inlet-water temperature differs appreciably from room temperature, an additional correction must be applied.

21. Calculate the *total heating values* by applying to the *observed heating values* the *correction for heat loss*, including the correction for the effect of difference between inlet-water temperature and room temperature, and the *correction for effect of atmospheric humidity*. The *average* of the *total heating values* found from all the series is then entered on the record.

The heating value thus found is, to within the limits of experimental error of the test, the total heating value of the gas.

Calculation in Case Inlet-water Temperature Differs from Room Temperature.—The correction to be applied in calculating the total heating value on account of the difference of temperature between room and inlet water will be different from that to be applied in calculating the net heating value, due to the fact that part of the change in the observed heating value is due to a difference in the amount of water vapor condensed in the calorimeter, a difference which is eliminated in the determination of the net heating value.

On page 526 are given the data from which to determine the amounts by which the total and the net heating values, calculated from the observed heating value as if the inlet water had been at room temperature, must be corrected on account of difference between inlet-water and room temperatures. For example, if the room temperature were 80°F. and the inlet-water temperature were 76°F., the total heating value, calculated in the usual way, is too high by 0.8 B.t u. per 1° difference; *i.e.*, the value is too high by $4 \times 0.8 = 3$ B.t.u. Similarly the net heating value is too high by $4 \times 0.4 = 2$ B.t.u.

The corrections calculated from page 526 may be applied without sensible error to heating values of illuminating gas of about 600 B.t.u. as determined with any of the flow calorimeters listed herein, except the Doherty calorimeter. The corrections for heat loss and for effect of atmospheric humidity must be made exactly as they would have been made if the inlet water had been at room temperature, and then the additional corrections, calculated from the table, must be applied to the results obtained for the total and net heating values. The corrections in the table are strictly applicable only for small differences between inlet-water temperature and room temperature. These data may also be used in correcting observations obtained when the inlet water is considerably below room temperature; but results obtained in this way will not be quite as reliable as those obtained when the temperature difference between room and inlet water is small. When this difference is large, the correction factor corresponding to the mean of the two temperatures should be used rather than the factor corresponding to room temperature.

The use of page 526 will be illustrated by the example below. Suppose the following data had been obtained with a flow calorimeter:

Room temperature = 70°F.
Humidity = 30 per cent.
Inlet-water temperature = 55°F.
Weight of water collected, W = 8.00 lb.
Corrected rise of temperature, T = 15.00°F.
Volume of gas, V = 0.2000 cu. ft.
Factor, F = 1.000.
Condensed water per cubic foot = 21.0 c.c.
Then:

$$\text{Observed heating value} = \frac{(W)\ 8.00 \times (T)\ 15.00}{(V)\ 0.2000 \times (F) 1.000} = 600 \text{ B.t.u.}$$

		Total	Net
Observed heating value	=	600	600
Correction for heat loss	=	+ 1	+ 1
Correction for atmospheric humidity (70°F., 40 per cent.)	=	+ 3	
Reduction to net = 21.0 × 2.3	=		− 48
Additional corrections for difference between inlet water and room temperatures:			
Correction for calculating total = 15 × 0.6	=	− 9	
Correction for calculating net = 15 × 0.4	=		− 6
Heating values	=	595	547

22. If it is desired to find the *net heating value*, proceed as follows:

(*a*) Subtract the *meter reading at the start* from the *meter reading at end* of *collection of condensate*. This difference gives approximately the number of cubic feet of gas (measured at *meter temperature* and under a pressure equal to that entered above as the *total gas pressure*) burned during the collection of the recorded *condensate* (*cc*). Multiply this difference, as found above, by the *factor*, F, to find the volume of the gas under standard conditions (*i.e.*, 60°F., 30 in. pressure), and divide the *condensate* (*cc*) collected by this product and enter the resulting quotient on the record as the *condensate per cubic foot* of gas measured at 60°F. and 30 in. pressure. Enter on the record the average, A, of the two values found as above from the two tests for condensed water collected.

The volume indicated by the meter (as given by the difference between the final and initial meter readings corresponding to the collection of condensate) is sufficiently accurate for the purpose of determining the net heating value unless the error of the meter is large (over 1 per cent.); thus, if the meter reading is in error by 2 per cent., the resulting error in the net heating value will be about 1 B.t.u. for an ordinary illuminating gas of about 600 B.t.u.

The two values obtained for condensate per cubic foot (60°, 30 in.) should not differ from one another by as much as 5 per cent. If the difference attains 5 per cent., it indicates that the drainage of the condensed water was not sufficiently regular to justify calculation of net heating values from the data obtained.

(*b*) Enter on the record the *average* of the *observed heating values.*

(*c*) Enter on the record the *correction for heat loss*, as already used in calculating the total heating value.

(*d*) Multiply the *condensate per cubic foot* of the gas (measured at 60°F. and 30 in.), entered opposite A in the record, by 2.3 and enter the product thus found as the *reduction to net.*

The amount of heat required for the vaporization, at the usual room temperatures, of 1 c.c. of water is about 2.3 B.t.u. The reduction to net as calculated above, therefore, represents the amount of heat imparted to the calorimeter by the condensation of the water collected.

HEAT VALUE TEST RECORD

Place, Bureau Stds. Date, Oct. 8, 1913 Time, 10 a.m. Gas, Gtwn.
Calorimeter No. J1209 Meter No. 6312 Thermometer No.: Inlet, 5781A Outlet, 5781B
Gas line purged Yes Meter adjusted Yes Water valve, 65 Damper, closed
Differential therm. corr'n. det'd. Oct. 1, 1913 Last meter calibration Sept. 4, 1913 (date)

	Start	End
Temp. of barometer	68°	68°
Barometer reading	29.52	29.51
Certif. corr'n	−0.01	
Temp. corr'n	−0.10	
Corr'd. barom. height	29.40	
Pressure at meter (inches of water)	1.5	
Equiv. (inches of mercury)	0.11	
Total gas pressure	29.51	
Meter therm. reading	68.1	68.3
Certif. corr'n	−0.3	
Meter temp	67.9	
Reduction factor F	0.963	
Psychrometer wet bulb	53.0	53.0
Psychrometer dry bulb	68.0	68.5
Humidity	35%	
Temp. of products	69	
Time of 1 meter rev.	52″	
Equiv. rate (cu. ft. per hr.)	6.9	

Condensed Water Collected	Test 1	Test 2
Meter reading: start	18.1	19.4
Meter reading: end	19.1	20.4
Condensate (cc)	21.6	21.2
Condensate per cu. ft. (60° 30 in.)	22.3	21.9
Average A	22.1	

Net Heating Value	
Observed heat value average	644
Corr'n. for heat loss	+ 1
Reduction to net ($A \times 2.3$)	−51
Net heating value	594

Certified as correct,
E. F. M.
Observer.

	Series No. 1		Series No. 2		Series No. 3	
	Inlet	Outlet	Inlet	Outlet	Inlet	Outlet
Preliminary	67.94	86.54	67.93	86.40	67.90	86.40
		50		45		45
Used in averaging		50		49		48
		43		40		40
		36		43		39
		43		47		43
		54		50		49
	67.93	49	67.98	50	67.90	52
		50		41		50
		50		40		40
		47		48		42
		40		38		45
Supplementary		49		41		46
	67.93	36	67.92	41	67.90	47
Average	67.93	86.46	67.93	86.45	67.90	86.45
Certificate corr'n	−18	−25				
Differential corr'n		−02	−18	−19	−18	−19
Emergent stem corr'n.		+08				
Corrected temp.	67.75	86.27	67.75	86.26	67.72	86.26
Temp. rise T	18.52		18.51		18.54	
Water heated W	6.74		6.74		6.72	
No. of rev. of meter	2					
Meter calib. 1 rev. =	0.1006					
Gas volume V	0.2012					
Observed heating value $\frac{W \times T}{V \times F}$	644		644		643	
Corr'n. for heat loss	+1					
Corr'n. for atmos. humid.	+4					
Total heating value	649		649		648	
Average	649 (B.t.u. per cu. ft. (60°, 30 in.)					

(*e*) To the *observed heating value* add the *correction for heat loss*, including the correction taken from page 526 for the effect of difference between inlet-water temperature and room temperature, and subtract the number derived as explained above, representing the *reduction to net*, and enter the result as the *net heating value*.

The heating value thus found is, to within the limits of experimental error of the test, the net heating value of the gas.

It is shown in *Tech. Paper* **36** that determinations of net heating values are not affected, provided combustion is complete, either by variations in the amount of air passing through the calorimeter or by variations in atmospheric humidity.

The observer making the test should sign the record as evidence that, to the best of his knowledge, the observations have been carried out as directed and that the computations have been correctly made. The signing of the record should not be regarded as a perfunctory operation and should not be done until the observer has satisfied himself by careful and critical inspection and review and by check computations that the results set forth are reliable.

Emergent Stem Corrections to Readings of Outlet-water Thermometers for Different Immersions of Thermometers in Calorimeter. (Bureau of Standards Circular 48.)

(Table applicable when temperature of inlet water is approximately equal to room temperature)

	Temperature rise of water, °F.	Temperature of room					
		50°	60°	70°	80°	90°	100°
Thermometer immersed to 30°F.	10	+0.02	+0.03	+0.04	+0.05	+0.05	+0.06
	15	+0.04	+0.05	+0.06	+0.07	+0.09	+0.10
	20	+0.06	+0.07	+0.09	+0.11	+0.13	+0.15
Thermometer immersed to 40°F.	10	+0.01	+0.02	+0.03	+0.03	+0.04	+0.05
	15	+0.03	+0.04	+0.05	+0.06	+0.08	+0.09
	20	+0.04	+0.05	+0.07	+0.09	+0.11	+0.12
Thermometer immersed to 50°F.	10	+0.01	+0.01	+0.02	+0.03	+0.04	+0.05
	15	+0.02	+0.03	+0.04	+0.05	+0.07	+0.08
	20	+0.02	+0.04	+0.06	+0.07	+0.09	+0.11
Thermometer immersed to 60°F.	10	+0.00	+0.01	+0.02	+0.02	+0.03	+0.04
	15	+0.00	+0.01	+0.03	+0.04	+0.05	+0.06
	20	+0.00	+0.02	+0.04	+0.05	+0.07	+0.09

This table is not applicable if the emergent portion of the stem includes an enlargement in the capillary.

Instead of using the above table, it will probably be somewhat more convenient to make out a stem-correction table for the particular outlet-water thermometer that is to be used with the calorimeter, the data for this separate stem-correction table being interpolated from the above table.

Suppose, for example, the outlet-water thermometer to be used was one that was immersed to the 30°F. mark on the scale, and a stem-correction table were wanted for an 18°F. rise in temperature, then from the above table we obtain the following stem-correction table:

STEM CORRECTION FOR OUTLET-WATER THERMOMETER No. —

(Table applicable when inlet water is approximately at room temperature, when thermometer is immersed to the 30°F. mark, and when the temperature rise is approximately 18°F.)

Inlet-water temperature, °F.	Stem correction, degrees	Inlet-water temperature, °F.	Stem correction, degrees
50	0.05	80	0.09
60	0.06	90	0.11
70	0.08	100	0.13

In the same way a table could be made out for any outlet-water thermometer by interpolation in the general table. The table so prepared would apply for the particular point to which the thermometer was immersed and for the particular rise in temperature with which the observer had chosen to work.

Corrections for Difference Between Inlet-Water Temperature and Room Temperature

[In this table are given the data from which to determine the amounts by which the total and the net heating values, calculated from the observed heating value as if the inlet water had been at room temperature, must be corrected on account of any difference in temperature between inlet water and room. The correction calculated from this table may be applied without sensible error to heating values of illuminating gas of about 600 B.t.u. as determined with any of the flow calorimeters except the Doherty calorimeter. The correction is added if the inlet water is warmer than the room; subtracted if the inlet water is colder. See page 522 for an example of method of application of these data to the calculation. In calculating the observed heating value, the stem corrections to both the inlet and outlet water thermometers must be taken into account when the inlet-water temperature differs from room temperature.]

Room temperature, °F	Corrections in B.t.u. per 1°F.	
	For calculating total heating value	For calculating net heating value
50	0.5	0.4
60	0.6	0.4
70	0.7	0.4
80	0.8	0.4
90	0.9	0.5
100	1.0	0.5

Correction Factors for Gas Volume. (From Bureau of Standards Circular 48.)

(Multiply observed volume of saturated gas by factor to correct to volume of saturated gas at 30 in. of mercury pressure (32°F.) and 60°F.[1])

Temperature, °F.	Total gas pressure—inches of mercury												
	28.0	28.1	28.2	28.3	28.4	28.5	28.6	28.7	28.8	28.9	29.0	29.1	29.2
99	0.825	0.828	0.831	0.834	0.837	0.840	0.844	0.847	0.850	0.953	0.856	0.859	0.863
98	0.828	0.831	0.834	0.838	0.841	0.844	0.847	0.850	0.853	0.856	0.860	0.863	0.866
97	0.831	0.834	0.838	0.841	0.844	0.847	0.850	0.853	0.856	0.860	0.863	0.866	0.869
96	0.834	0.838	0.841	0.844	0.847	0.850	0.853	0.856	0.860	0.863	0.866	0.869	0.872
95	0.837	0.841	0.844	0.847	0.850	0.853	0.856	0.860	0.863	0.866	0.869	0.872	0.876
94	0.841	0.844	0.847	0.850	0.853	0.856	0.860	0.863	0.866	0.869	0.872	0.876	0.879
93	0.844	0.847	0.850	0.853	0.856	0.860	0.863	0.866	0.869	0.872	0.876	0.879	0.882
92	0.847	0.850	0.853	0.856	0.860	0.863	0.866	0.869	0.872	0.875	0.879	0.882	0.885
91	0.850	0.853	0.856	0.859	0.862	0.866	0.869	0.872	0.875	0.878	0.882	0.885	0.888
90	0.853	0.856	0.859	0.862	0.866	0.869	0.872	0.875	0.878	0.882	0.885	0.888	0 891
89	0.856	0.859	0.862	0.865	0.868	0.872	0.875	0.878	0.881	0.885	0.888	0.891	0.894
88	0.859	0.862	0.865	0.868	0.872	0.875	0.878	0.881	0.884	0.888	0.891	0.894	0.897
87	0.861	0.865	0.868	0.871	0.874	0.878	0.881	0.884	0.887	0.890	0.894	0 897	0.900
86	0.864	0.868	0.871	0.874	0.877	0.880	0.884	0.887	0.890	0.893	0 897	0.900	0.903
85	0.867	0.870	0.874	0.877	0.880	0.883	0.887	0.890	0.893	0.896	0.900	0.903	0.906
84	0.870	0.873	0.876	0.880	0.883	0.886	0.890	0.893	0.896	0 899	0.902	0.906	0.909
83	0.873	0.876	0.879	0.883	0.886	0.889	0.892	0.896	0.899	0.902	0.905	0.909	0.912
82	0.876	0.879	0.882	0.885	0.889	0.892	0.895	0.898	0.902	0.905	0.908	0.911	0.915
81	0.878	0.882	0.885	0.888	0.891	0.895	0.898	0.901	0.904	0.908	0.911	0.914	0 918
80	0.881	0.884	0.888	0.891	0.894	0.897	0.901	0.904	0.907	0.910	0.914	0.917	0.920
79	0.884	0.887	0.890	0.894	0.897	0.900	0.904	0.907	0.910	0.913	0.916	0.920	0.923
78	0.887	0 890	0.893	0.896	0 900	0.903	0.906	0.910	0.913	0.916	0 919	0 923	0.926
77	0.889	0 892	0.896	0 899	0.902	0.906	0.909	0.912	0.916	0 919	0 922	0.925	0 929
76	0.892	0.895	0.898	0.902	0.905	0 908	0.912	0.915	0.918	0.921	0.925	0 928	0.931
75	0.894	0.898	0.901	0.904	0.908	0.911	0.914	0.918	0.921	0.924	0.928	0.931	0.934
74	0.897	0 900	0.904	0.907	0.910	0.914	0.917	0 920	0 924	0.927	0.930	0.933	0 937
73	0.900	0 903	0.906	0.910	0.913	0 916	0.920	0.923	0 926	0.930	0 933	0 936	0.939
72	0.902	0.906	0 909	0.912	0.916	0.919	0 922	0.926	0 929	0.932	0.936	0.939	0 942
71	0 905	0.908	0.912	0 915	0.918	0.922	0 925	0.928	0.931	0.935	0.938	0.941	0.945
70	0 907	0.911	0.914	0.917	0 921	0.924	0.927	0.931	0.934	0.937	0.941	0.944	0.947
69	0 910	0.913	0.917	0.920	0.923	0.927	0.930	0.933	0.937	0.940	0.943	0.947	0.950
68	0.912	0.916	0.919	0.922	0.926	0.929	0.932	0.936	0.939	0.942	0.946	0.949	0.952
67	0.915	0.918	0.922	0.925	0.928	0 932	0.935	0.938	0.942	0.945	0.948	0.952	0.955
66	0.917	0.921	0.924	0.928	0.931	0.934	0.938	0.941	0.944	0.948	0.951	0.954	0.958
65	0.920	0.923	0.927	0.930	0.933	0.937	0.940	0.944	0.947	0.950	0.954	0.957	0.960
64	0.922	0 926	0 929	0.932	0.936	0.939	0.943	0.946	0.949	0.953	0 956	0.959	0.963
63	0.925	0.928	0.932	0 935	0.938	0.942	0.945	0.948	0 952	0.955	0 959	0 962	0.965
62	0.927	0.931	0.934	0 937	0.941	0.944	0.948	0.951	0.954	0.958	0.961	0.964	0.968
61	0.930	0.933	0.936	0.940	0.943	0.947	0.950	0.953	0.957	0.960	0.964	0.976	0.970
60	0.932	0.936	0.939	0.942	0.946	0.949	0 953	0 956	0.959	0.963	0.966	0.969	0.973

[1] Formula used: Correction factor $= \frac{(P - E)(60 + 459.4)}{(t + 459.4)(30 - 0.5170)}$.

P = total gas pressure.
E = vapor pressure of water at $t°$.
t = temperature of gas (°F.).

CORRECTION FACTORS FOR GAS VOLUME—*Continued.*

(Multiply observed volume of saturated gas by factor to correct to volume of saturated gas at 30 in. of mercury pressure (32°F.) and 60°F.[1])

Temperature, °F.	Total gas pressure—inches of mercury												
	29.3	29.4	29.5	29.6	29.7	29.8	29.9	30.0	30.1	30.2	30.3	30.4	30.5
99	0.866	0.869	0.872	0.875	0.878	0.882	0.885	0.888	0.891	0.894	0.897	0.900	0.904
98	0.869	0.872	0.875	0.879	0.882	0.885	0.888	0.891	0.894	0.898	0.901	0.904	0.907
97	0.872	0.876	0.879	0.882	0.885	0.888	0.891	0.894	0.898	0.901	0.904	0.907	0.910
96	0.876	0.879	0.882	0.885	0.888	0.891	0.895	0.898	0.901	0.904	0.907	0.910	0.914
95	0.879	0.882	0.885	0.888	0.892	0.895	0.898	0.901	0.904	0.907	0.911	0.914	.917
94	0.882	0.885	0.888	0.892	0.895	0.898	0.901	0.904	0.907	0.911	0.914	0.917	0.920
93	0.885	0.888	0.892	0.895	0.898	0.901	0.904	0.908	0.911	0.914	0.917	0.920	0.923
92	0.888	0.891	0.895	0.898	0.901	0 904	0.907	0.911	0.914	0.917	0.920	0.923	0.927
91	0.891	0.894	0.898	0.901	0.904	0.907	0.910	0.914	0.917	0.920	0.923	0.926	0.930
90	0.894	0.898	0.901	0.904	0.907	0.910	0.914	0.917	0.920	0.923	0.926	0.930	0.933
89	0.897	0.901	0.904	0.907	0.910	0.914	0.917	0.920	0.923	0.926	0.930	0.933	0.936
88	0.900	0.904	0.907	0.910	0.913	0.916	0.920	0.923	0.926	0.929	0.933	0.936	0.939
87	0.903	0.907	0 910	0.913	0.916	0.920	0.923	0.926	0.929	0.932	0.936	0.939	0.942
86	0.906	0.910	0.913	0.916	0 919	0.922	0.926	0.929	0.932	0.935	0.939	0.942	0.945
85	0.909	0.912	0.916	0.919	0.922	0.925	0.929	0.932	0.935	0.938	0.942	0.945	0.948
84	0.912	0.915	0 919	0.922	0.925	0.928	0.932	0.935	0.938	0.941	0.945	0.948	0.951
83	0.915	0.918	0.922	0.925	0.928	0.931	0.934	0.938	0.941	0.944	0.948	0.951	0.954
82	0.918	0.921	0.924	0.928	0.931	0.934	0.937	0.941	0.944	0.947	0.950	0.954	0.957
81	0.921	0.924	0.927	0.930	0.934	0.937	0.940	0.944	0.947	0.950	0.953	0.957	0.960
80	0.924	0.927	0.930	0.933	0.937	0.940	0.943	0.946	0.950	0.953	0.956	0.960	0.963
79	0.926	0.930	0.933	0.936	0.940	0.943	0.946	0.949	0.952	0.956	0.959	0.962	0.966
78	0.929	0.932	0.936	0.939	0.942	0.946	0.949	0.952	0.955	0.959	0.962	0.965	0.968
77	0.932	0.935	0.938	0.942	0.945	0.948	0.952	0.955	0.958	0.961	0.965	0.968	0.971
76	0.935	0.938	0.941	0.944	0.948	0.951	0.954	0.958	0.961	0.964	0.968	0.971	0.974
75	0.937	0.941	0.944	0.947	0.950	0.954	0.957	0.960	0.964	0.967	0.970	0.974	0.977
74	0.940	0.943	0.947	0.950	0.953	0.957	0.960	0.963	0.966	0.970	0.973	0.976	0.980
73	0.943	0.946	0.949	0.953	0.956	0.959	0.963	0.966	0.969	0.972	0.976	0.979	0.982
72	0.945	0.949	0.952	0.955	0.959	0.962	0.965	0.969	0.972	0.975	0.978	0.982	0 985
71	0.948	0.951	0.955	0.958	0.961	0.965	0.968	0.971	0.975	0.978	0.981	0.985	0.988
70	0.951	0.954	0.957	0.961	0.964	0.967	0.971	0 974	0.977	0.981	0.984	0.987	0.991
69	0.953	0.957	0.960	0.963	0.967	0.970	0.973	0.977	0.980	0.983	0.987	0.990	0.993
68	0.956	0.959	0.962	0.966	0.969	0.973	0.976	0.979	0.983	0.986	0.989	0.993	0.996
67	0.958	0.962	0 965	0.968	0.972	0.975	0.979	0.982	0.985	0.989	0.992	0.995	0.999
66	0.961	0.964	0.968	0.971	0.974	0.978	0.981	0.984	0.988	0.991	0.995	0.998	1.001
65	0.964	0.967	0.970	0.974	0.977	0.980	0.984	0.987	0.990	0.994	0.997	1.001	1.004
64	0.966	0.970	0.973	0.976	0.980	0.983	0.986	0.990	0.993	0.996	1.000	1.003	1.007
63	0.969	0.972	0.975	0.979	0.982	0.986	0.989	0.992	0.996	0.999	1.002	1.006	1.009
62	0.971	0.975	0.978	0.981	0.985	0.988	0.992	0.995	0.998	1.002	1.005	1.008	1.012
61	0.974	0.977	0.980	0.984	0.987	0.991	0.994	0.997	1.001	1.004	1.008	1.011	1.014
60	0.976	0.980	0.983	0.986	0.990	0.993	0.997	1.000	1.003	1.007	1.010	1.014	1.017

[1] Formula used: Correction factor $= \frac{(P - E)(60 + 459.4)}{(t + 459.4)(30 - 0.5170)}$.

P = total gas pressure.
E = vapor pressure of water at t°.
t = temperature of gas (°F.).

CORRECTIONS, IN B.T.U., TO BE APPLIED TO OBSERVED HEATING VALUES IN CALCULATING TOTAL HEATING VALUES OF ILLUMINATING GAS
(ABOUT 600 B.T.U.)

(The tabular corrections are applicable when inlet water, air, gas, and products are all at approximately the same temperature and when the calorimeter is operated at normal rate of gas consumption)

Temperature of room, etc., °F.	Relative humidity of air									
	10 per cent.	20 per cent.	30 per cent.	40 per cent.	50 per cent.	60 per cent.	70 per cent.	80 per cent.	90 per cent.	100 per cent.
40	+ 2	+ 2	+ 1	+1	+1	+1	0	0	0	−1
45	+ 2	+ 2	+ 2	+1	+1	+1	0	0	0	−1
50	+ 3	+ 3	+ 2	+2	+1	+1	0	0	0	−1
55	+ 3	+ 3	+ 3	+2	+1	+1	+1	0	0	−1
60	+ 4	+ 4	+ 3	+2	+2	+1	+1	0	0	−1
65	+ 5	+ 4	+ 4	+3	+2	+2	+1	0	−1	−1
70	+ 6	+ 5	+ 4	+3	+3	+2	+1	0	−1	−2
75	+ 7	+ 6	+ 5	+4	+3	+2	+1	+0	−1	−2
80	+ 8	+ 7	+ 6	+5	+4	+3	+1	0	−1	−2
85	+10	+ 9	+ 7	+6	+4	+3	+2	0	−1	−3
90	+12	+10	+ 9	+7	+5	+4	+2	0	−2	−3
95	+14	+12	+10	+8	+6	+4	+2	0	−2	−4

CORRECTIONS, IN B.T.U., TO BE APPLIED TO OBSERVED HEATING VALUES IN CALCULATING TOTAL HEATING VALUE OF NATURAL GAS
(ABOUT 1000 B.T.U.)

(The tabular corrections are applicable when inlet water, air, gas, and products are all at approximately the same temperature and when the calorimeter is operated at normal rate of gas consumption)

Temperature of room, etc., °F.	Relative humidity of air									
	10 per cent.	20 per cent.	30 per cent.	40 per cent.	50 per cent.	60 per cent.	70 per cent.	80 per cent.	90 per cent.	100 per cent.
40	+ 4	+ 3	+ 3	+ 2	+ 2	+1	+1	0	0	−1
45	+ 4	+ 4	+ 3	+ 3	+ 2	+1	+1	0	0	−1
50	+ 5	+ 5	+ 4	+ 3	+ 3	+2	+1	0	0	−1
55	+ 6	+ 6	+ 5	+ 4	+ 3	+2	+1	0	−1	−1
60	+ 8	+ 7	+ 6	+ 4	+ 3	+2	+1	0	−1	−2
65	+ 9	+ 8	+ 7	+ 5	+ 4	+3	+2	0	−1	−2
70	+11	+ 9	+ 8	+ 6	+ 5	+3	+2	+1	−1	−2
75	+13	+11	+10	+ 8	+ 6	+4	+3	+1	−1	−3
80	+15	+13	+11	+ 9	+ 7	+5	+3	+1	−1	−3
85	+18	+16	+13	+11	+ 9	+6	+4	+1	−2	−4
90	+21	+19	+16	+13	+10	+7	+4	+1	−2	−5
95	+25	+22	+19	+15	+12	+8	+5	+1	−2	−6

Approximate English-Metric Equivalents

1 inch = 2.540 centimeters.
1 yard = 0.9144 meter.
1 cubic inch = 16.39 cubic centimeters.
1 cubic foot = 0.0283 cubic meter.
1 liquid ounce = 29.57 cubic centimeters.
1 liquid quart = 0.9463 liter.
1 grain = 0.0648 gram.
1 ounce avoirdupois = 28.35 grams.
1 pound avoirdupois = 0.4536 kilogram.
1° Fahrenheit = 5/9 degree Centigrade.
1 British thermal unit = 0.2520 calorie.
1 B.t.u. per cubic foot = 8.89 calories per cubic meter.
1-inch water pressure = 1.87-mm. Hg pressure.

Corrections for the Reduction of Barometric Heights to Standard Temperature (32°F.)

(In the table are given, for barometric heights of 20, 25, and 30 in., the amounts, in inches, to be subtracted from the observed readings of a barometer with brass scale, to reduce the heights, observed at various temperatures, to inches of mercury at 32°F.)

Temperature, °F.	Observed barometric height, inches		
	20	25	30
40	0.02	0.03	0.03
50	0.04	0.05	0.06
60	0.06	0.07	0.09
70	0.08	0.09	0.11
80	0.09	0.12	0.14
90	0.11	0.14	0.17
100	0.13	0.16	0.19

Record Sheet for Gas Calorimeter Readings

No......

GAS CALORIMETER READINGS

....

Place of Test..... Date.....

	Time	Time	Time
Kind of gas.....			
Calorimeter, kind.....			
Barometer reading..... *			
Temperature of gas..... *			
Correction factor.....			
Temperature of atmosphere.....			
Temperature of exhaust.....			
Condensed water collected.....			
Time one revolution of meter.....			
Gas consumed during test..... *			
Rate of combustion..... *			
Weight of water..... *			
Per cent. atmospheric humidity.....			

Temperatures of water		Inlet	Outlet	Inlet	Outlet	Inlet	Outlet
	1						
	2						
	3						
	4						
Read at least ten times during	5						
two revolutions of meter hand	6						
	7						
	8						
	9						
	10						
Average temperature.....							
Thermometer correction.....							
Actual temperature.....							
Rise in temperature..... *							

B.t.u. recorded.....			
Correction for atmospheric humidity...			
B.t.u. corrected.....			

In using heating value computer, take figures indicated by asterisk*

.....Observer

Heating Values of Various Gases (B.t.u. per Cu. Ft. at 60°F. and 30 In. of Mercury)

Hydrogen	326.2
Carbon monoxide	323.0
Ethylene	1588.0
Propylene	2347.0
Butylene	3099.0
Acetylene	1476.0
Benzene	3807.0
Coal	600.0
Blue water-gas	340.0
Blast-furnace	108.0
Producer	165.0
Blau	1700.0
Pintsch	1500.0
Liquefied petroleum gas	2200.0

Laboratory Gas Meters. (From Bureau of Standards Circular 48.)

(*a*) **Description.**—Laboratory gas meters, which are intended for precise measurement of gas volumes, are commonly of the wet-meter type, and are usually made with capacities of $\frac{1}{10}$ or $\frac{1}{12}$ cu. ft. or of 3 liters for one revolution of the measuring drum; $\frac{1}{20}$ cu. ft. meters are also made for inspection work.

A detailed description of the construction and mode of operation of laboratory gas meters, together with an account of an investigation of the sources of error to which such meters are liable, and the accuracy attainable in their use, may be found in *Technologic Paper* No. **36** of the Bureau of Standards.

The mode of operation of such meters will be described with reference to the diagrammatic sketch of Fig. 138. The meter consists of an outer cylindrical casing which is filled with water to the level indicated. Within this casing is the measuring drum rotating on a horizontal axis. This axis projects through the front of the meter casing, passing through a stuffing box. The bearing at the other end of the axis, not shown in the figure, is entirely under water.

Outside of the stuffing box the end of the axis of the drum is connected to the indicating mechanism of the meter. It is preferable to have the principal hand (the long hand) of the meter mounted directly upon the axis of the drum and in such a way that the hand can not slip relative to the drum or axis. The smaller hands of the meter, which indicate multiples of the quantities indicated by the principal hand, are connected to the axis of the drum by means of a system of gear wheels.

The measuring drum has five compartments, four of which are made by dividing the cylindrical portion by means of suitable partition walls. These four compartments are so shaped that the openings of each compartment, one on the front and one on the back of the drum, are nearly radial and make an angle of about 160° with each other. The fifth compartment

covers the whole of the back face of the measuring drum, and opens into each of the four other compartments.

The mode of operation of such a meter may be understood by following one of the four compartments of the drum through a complete revolution, starting with the compartment at its lowest position, when it is full of water and both the openings are under water.

The gas, passing through the inlet tube enters the back compartment and by its pressure on the inclined faces of the partition walls of the other compartments, causes the drum to revolve. The opening at the rear of the compartment which is under water emerges, allowing the gas to enter the compartment, while the contained water is discharged through the other

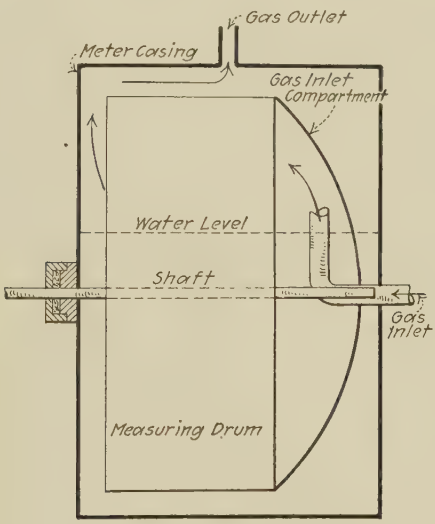


FIG. 138.—Section of experimental gas meter.

openings along the axis and at the front. As the drum continues to revolve, the compartment gradually becomes filled with gas. However, before the opening at the front emerges from the water the one at the back dips into and is closed by the water, sealing off momentarily a certain volume of gas. As the drum now continues to revolve, the opening at the front emerges from the water and the gas escapes into the outer casing, being displaced by the water entering through the large opening along the axis and the radial opening at the back, the process continuing until the compartment is again entirely submerged. The gas is discharged from the meter casing through the outlet connection.

During each revolution, each compartment delivers the amount of gas it contained at the instant it was sealed off, an amount which will evidently depend on the position of the water level.

(*b*) **Choice of Meter.**—There is little that can be said that will aid the purchaser in the choice of a meter, as the working parts are concealed and wearing qualities are not easily determined by inspection. All the wet meters furnished with the gas calorimeters listed in *circular* **48,** with one exception, were practically identical in essential details of design, and differed principally in the materials of construction and in the grade of workmanship, factors on which the satisfactory operation of the meters is largely dependent. Important features are as follows:

1. The leveling device usually consists of three leveling screws on tripod base with two levels at right angles, or a circular level, or a plumb bob on the meter.

2. Provision should be made for adjustment of water level in the meter to a fixed mark, either with a gage glass, a sight box (which is an extension of the meter casing having front and back faces of glass), or an automatic overflow. The gage glass and the sight box are equally satisfactory, but the automatic overflow, as exemplified in the Elster meter furnished with the Junkers calorimeters, was found to be unsatisfactory. The inside diameter of the gage glass should be not less than $\frac{3}{8}$ in. and the connecting tubes between the gage glass and the meter should be so large as to avoid the possibility of the passage being obstructed. The mark to which the water level is set must be of such form that accurate settings can be made; a simple pointer on the outside of the gage glass is not satisfactory.

3. The use of materials that will not give trouble due to corrosion is important.

4. Well-made bearings, stuffing boxes, etc., and accessibility of stuffing box for repacking are essential.

5. Proper shaping of the measuring compartments of the drum to give a fair degree of proportionality of the graduated scale and to avoid undue fluctuations of the water level during the rotation of the drum, is, of course, very necessary.

6. Provision should be made for emptying and cleaning the meter and for running in and withdrawing water in adjusting the water surface, as well as provision for draining the "dry well" at the meter inlet.

7. An enamel dial is preferable, without glass face plate. While the latter does not increase the error due to parallax, it reflects light and sometimes increases the difficulty of reading.

8. In $\frac{1}{10}$ and $\frac{1}{12}$ cu. ft. meters the subdivision of the dial is usually into 100 parts, each tenth graduation being numbered and being somewhat longer than the intermediate graduations; it would be a convenience to also lengthen every fifth graduation. An auxiliary dial giving the rate of gas delivered from readings of the meter dial one minute apart is also provided on some meters, as well as suitable small dials on the face of the meter to register the number of revolutions of the meter index.

9. The large hand should extend to the middle of the graduations, with its end close to the dial.

10. A thermometer should be provided, with its bulb immersed in the water in the meter.

11. A well-made meter in proper adjustment, with the stuffing box packing not too tight, should operate on a small and constant back pressure,

so that the calibration will be practically independent of the rate up to rates of about 10 cu. ft. per hour.

(*c*) **Adjustment of Meter.**—Before proceeding with the calibration, the meter must be carefully adjusted as follows:

1. Level the meter with the aid of the levels or the plumb bob mounted on the meter. Care should be taken to insure that the levels are not loose nor in such condition that they can be easily displaced relative to the meter.

2. Adjust the index of the meter relative to the drum in the following manner: It will be observed that there is considerable fluctuation of the water level in the gage glass four times during each revolution of the index, these fluctuations occurring each time that a compartment is sealed off. The index should be so set relative to the drum that the index will pass through the zero position midway between two such fluctuations of water level; *e.g.*, if the dial is divided into 100 divisions, the index should be at 12.5, 37.5, 62.5, and 87.5 approximately at the times these fluctuations in water level occur. Unfortunately meters are usually made with slip hands, so that this adjustment must be frequently verified by the user.

3. Adjust the surface of the water in the meter. The water in the meter should be saturated with the medium used in testing, by passing 2 or 3 cu. ft. of the gas or the air through the meter before this adjustment is made; and enough water should be used to bring its level somewhat above the fixed mark to which it is to be adjusted. Pass enough gas through the meter to bring the long hand to the zero position and then disconnect the tubing leading to the inlet, thus leaving both inlet and outlet open to the air. To avoid displacing the meter, it is better to disconnect not at the meter, but at the distant end of the inlet tube. Drain off the water until the lower boundary of the meniscus in the gage glass or in the sight box coincides with the fixed mark. The meter should then be again connected to the gas supply, run one or more revolutions, again disconnected, and the adjustment verified.

An error of $\frac{1}{16}$ in. in the setting of the water surface, a similar displacement of the fixed mark, or an equivalent error in the leveling of the meter will cause an error of about 1 per cent. in the indications. It is evident therefore that the leveling of the meter and the adjustment of the water surface should be done with *great care.*

(*d*) **Calibration of Meter.**—For the calibration of the laboratory wet gas meter one may use a fractional cubic-foot bottle, a cabinet-type or an immersion-type cubic-foot bottle, a meter prover, or a calibrated meter; or the meter may be tested by direct weighing of the water displaced from a suitable container by the gas, or displacing the gas which passes through the meter during one or more revolutions of the drum. For any one of these methods either air or gas may be used as the testing medium if care is taken to saturate the water with the air or gas before the test and to prevent the water from giving up any of the dissolved gas during the test. The water in the test apparatus may be quickly saturated by bubbling gas through it and the water in the meter is saturated by the passage of 1 or 2 cu. ft. of gas. Except when it is desired to calibrate a meter containing the same water that has previously been used in it, it is generally more convenient to test with air than with gas.

In the calibration of the meter an accuracy of 0.2 or 0.3 per cent. or better should be secured if possible. If the highest accuracy obtainable in the use of a wet meter is to be obtained, it must be calibrated in place after adjustment at the time when it is to be used. However, this procedure is not generally necessary, since the probable accuracy of readjustment of the meter is usually sufficient when care is taken, and the procedure is often inconvenient. (See "interpretation of results," page 539.)

If considerable resistance is offered to the motion of the meter drum, as evidenced by a considerable drop of gas pressure between inlet and outlet, then the capacity of the drum per revolution will be appreciably different at different rates of gas passage. For this reason a meter which shows a difference of pressure between inlet and outlet of more than 0.1 or 0.2 of an inch of water pressure should be tested when running at the speed at which it is to be used. A meter showing a difference of pressure of 0.1 in. or less may generally be tested at any speed less than the one at which it is to be used without introducing serious error in the result.

Tests of laboratory meters can be successfully made with either the cabinet-type or the immersion-type cubic-foot bottle at any speed at which such meters would ordinarily be used. The precautions and operating method will be evident from the following section, describing the use of a fractional cubic-foot bottle and from the directions given for the calibration of meter provers. When using either type of bottle, the air should remain in the bottle until there is no doubt that it is fully saturated with water vapor and at the temperature of the bottle. In using the immersion-type bottle care must be taken to prevent changes of temperature due to evaporation of the water from the surface of the bottle as described under the section on "calibration of meter provers."

Using a meter prover which has been calibrated, the test of a laboratory meter may be performed at the speed at which the meter is to be used; but it is necessary to exercise great care in securing nearly constant temperature of the air inside and around the prover bell. In this respect the covered-bell prover has marked advantages. If the test is carried out with the air in the prover at the ordinary pressure of 1½ in. of water, the speed of the meter should be regulated by stopping down the outlet of the meter so that the rate will be the same as that while the meter is being used. In such tests a sufficient quantity of air should be discharged through the meter to permit reasonable accuracy in the reading of the prover; with the ordinary 5-cu. ft. prover at least 2 cu. ft. of gas should be used in order to secure an accuracy of 0.1 per cent.

The method of comparison of a laboratory meter with another calibrated meter is described in detail in *Technologic Paper* No. **36** of the Bureau of Standards. If a meter which requires a small differential pressure for its calibration has been calibrated by the use of a fractional cubic-foot bottle, this can then be used for testing other meters at various speeds even though the other meters require considerable pressure for their operation at these higher rates.

The method of testing, in which the water which displaces or is displaced by the gas is weighed, is described in detail in *Technologic Paper* No. **36** of the Bureau of Standards.

(*e*) **Calibration with Fractional Cubic-Foot Bottle.**—The ordinary fractional cubic-foot bottle can not be conveniently used to test meters at a speed as great as 5 or 6 cu. ft. per hour; it is, however, very convenient for testing those meters which may be tested at a lower speed. Since many of the modern American-made meters are included in this class, the fractional cubic-foot bottle method is much used.

The fractional cubic-foot bottle is a metal container terminating at the top and at the bottom in glass tubes. The bottle is made so that the volume included between two gage marks on the glass tubes is equal to $\frac{1}{10}$ (or $\frac{1}{12}$) cubic foot. The upper outlet of the bottle is closed by a three-way stopcock, by means of which the bottle may be connected either to a supply of gas or to the meter to be tested. The lower outlet of the bottle is connected by flexible tubing to a movable reservoir. Enough water is put into the system so that when the movable reservoir is set on the lower of two adjustable shelves, the water level in the bottle will be at the lower gage mark; while when the movable reservoir is set on the upper shelf, the water level in the bottle will be at the upper gage mark. To facilitate maintenance of the proper temperature, the bottle may be surrounded by a small tank filled with water at the temperature of the water in the meter.

For the calibration of a $\frac{1}{10}$-cu. ft. meter a $\frac{1}{10}$-cu. ft. bottle, and for the calibration of a $\frac{1}{12}$-cu. ft. meter the $\frac{1}{12}$-cu. ft. bottle would, of course, be chosen.

Manipulation.—After the water in both meter and bottle are saturated with gas or air and the temperature of both instruments and the contained water is adjusted to be equal to room temperature, the meter is leveled and the water surface in it adjusted. The amount of water in the bottle and the height of the supports for the leveling tank are also adjusted to bring the water to the proper graduations at each position of the tank. The test is then carried out as follows:

1. Connect one branch of the three-way stopcock of the bottle to the gas supply and the other branch to the meter inlet. Connect a burner provided with a pilot light to the outlet of the meter.

2. Starting with the bottle filled with water, displace the water with gas.

3. Test for gas leaks. Force the gas in the bottle through the meter, but before all the gas has been discharged from the bottle shut off the gas beyond the meter. Put a block about 2 in. high on the upper adjustable shelf and set the movable reservoir on this, and allow a sufficient quantity of gas to escape through the meter so that the water in the bottle is visible in the upper glass tube. The pressure on the gas will then be equivalent to at least 2 in. of water. Then observe whether, during a period of five minutes, there is any appreciable displacement of the water level in the bottle.

4. Again fill the bottle with gas and force enough gas through the meter to bring the index to the zero position.

5. Fill the bottle with gas. Since the gas is supplied under pressure, it is necessary after the bottle is filled to disconnect it from the gas supply and allow the surplus gas to escape into the air (not through the meter). The water level in the bottle should now be at the gage mark.

6. Note and record the temperature of the room, of the meter, and of the water in the movable reservoir, and the reading of the meter.

7. Raise the movable vessel to the upper adjustable shelf, displacing the gas contained in the bottle through the meter. When the water level in the bottle has come to rest at the upper gage mark, read and record the reading of the meter.

8. Refill the bottle and displace the gas through the meter, performing the operation five or six times, recording temperatures and meter readings each time.

Computations.—1. The volume of the bottle may be so accurately adjusted that there should be no necessity for applying a correction for the error in the volume of the bottle. The change in the volume of the bottle, due to the thermal expansion of the metal, may be neglected.

2. The gas in the bottle is measured at atmospheric pressure, but while this gas is forced through the meter the pressure is increased slightly, and in the meter the gas is measured at this increased pressure, with the result that the volume indicated by the meter is smaller than it would have been if measured at the same pressure in the meter as in the bottle. Usually the increase of pressure will be *only a few tenths of an inch of water*, so that the error introduced will be *negligible*. It is well, however, to connect a U gage to the line leading from the bottle to the meter so as to measure the pressure existing while the meter is running. If the pressure was found to be equivalent on the average to *1 in. of water* the volume indicated by the meter would have to be increased by 0.2 per cent. It is preferable, however, to make the manipulations so that the correction will be small enough to be negligible.

3. If the water in the bottle and that in the meter are at different temperatures, then the gas will successively acquire these temperatures and tend to expand or contract in passing from the bottle to the meter, depending upon whether the water of the meter is at a higher or lower temperature than that in the bottle. Correction for such difference in temperature must therefore be applied to the results obtained in order to obtain a correct comparison of the meter and the bottle. These corrections may be made by the use of the formulas already given (page 527); or the volume under each condition of measurement may be reduced to standard conditions, using the correction factors given on page 527. However, if the difference in temperature between the meter and the bottle is very small, it is possible to correct for this by increasing the volume indicated by the meter by 0.3 per cent. for each 1°F. that the meter temperature is below the bottle temperature. This last procedure should be adopted for only small differences of temperature.

4. The calculations should be made separately for each bottle full of gas displaced through the meter and the results averaged. While the same result would be obtained by taking only the initial and final readings, no check would be provided on the accuracy of the test if this were done; and possibly a false impression would be created as to the accuracy attained.

Example. Suppose the following observations were made:

Temperature of room	70.0°F.
Temperature of bottle	70.5°F.
Temperature of meter	69.5°F.
Gas pressure while meter is running (inches of water)	1.4
Initial meter reading	15.1993
Final meter reading	15.2996

The simple method of correction would then be as follows:

Volume indicated by meter	0.1003
Correction for pressure	+3
Correction for temperature	+3
Volume indicated by meter per 0.1 cu. ft. of gas	0.1009

Therefore, the meter delivers 0.0991 cu. ft. per revolution.

In this example significant differences in temperature and pressure were assumed in order to illustrate the method of making the corrections. In an actual test it is desirable, and generally possible, to make these differences so small that no correction is necessary.

Alternate Method.—If a supply of gas at constant pressure—*e.g.*, from a gas holder—is available, there are some advantages in using the method of displacing the gas through the meter into the bottle. The procedure outlined may be readily modified so as to be suitable for use in this method of calibration. The principal modification would be in making the test for gas leaks. This test should be made with the bottle filled with gas and the indication of leakage would be given by the motion of the meter index.

(*f*) **Interpretation of Result.**—The result obtained by the calibration of the meter with the particular adjustments which it had at the time of the test is the one to be used for the meter on the day that the calibration is carried out, provided, of course, the water level, etc., of the meter are not readjusted. However, when the meter is used after it has been releveled and the water level readjusted, then the average of all recent calibrations should be used, since it is more probable that this average corresponds to the adjustment made. A number of calibrations for the meter when set up and adjusted should be made at different times; and unless a progressive change in the meter is indicated, the average of all the results should be used as best representing the calibration.

Analysis of Casinghead Gas for Gasoline Content. (Hill's "The Production of Natural Gas in 1913.")

Analysis No.	Date of analysis	Location of field	Absorption heavy hydrocarbons, per cent.	Carbon dioxide	Oxygen	Nitrogen	Specific gravity	Combustion ratios			R	R^1	Gallons per thousand cubic feet of gas
								Contraction	CO_2	CO_2			
2500	Apr. 2, 1913	Calgary, Alberta, Can.	15.0	None	None	None	0.67	2.17	1.26	2.37	1.72	0.390	Dry
2430	Jan. 25, 1913	Electra, Tex.	50.0	None	None	None	1.12	2.56	2.03	3.59	1.26	0.889	3.5
2242	May 20, 1912	Oilfields, Cal.	36.0	....	0.70	3.8	0.79	2.21	1.13	2.34	1.96	0.403	1.0
2747	Nov. 17, 1913	Casper, Wyo.	45.1	None	None	None	1.04	2.57	1.98	4.54	1.29	0.806	3.0
2748	Nov. 17, 1913	Casper, Wyo.	35.5	None	None	None	0.94	2.47	1.72	3.19	1.44	0.653	2.0
1477	Feb. 23, 1911	Glen Pool, Okla.	62.5	4.7	0.80	3.0	1.16	2.62	2.15	3.77	1.22	0.951	4.0
2181	Jan. 2, 1912	Childers, Okla.	79.3	None	None	None	1.37	2.89	2.55	4.43	1.13	0.120	5.5
2065	July 17, 1911	Bremen, Ohio.	25.0	None	0.30	(a)	0.67	2.09	1.10	2.19	1.98	0.338	Dry
D45	Oct. 17, 1911	Cherryvale, Kans.	21.2	0.95	None	None	0.60	2.03	1.01	2.04	2.00	0.300	Dry
2533	Apr. 28, 1913	Titusville, Pa.	49.2	1.50	None	None	0.90	2.48	1.64	3.13	1.51	0.596	2.0
1632	Mar. 18, 1911	Sistersville, W. Va.	45.0	0.70	6.90	28.3	1.52	2.82	2.89	4.72	0.976	1.560	Air-free
1186	Jan. 26, 1910	Sistersville, W. Va.	62.0	None	2.50	(a)	1.23	2.72	2.24	3.95	1.21	1.020	4.5
1494	Feb. 23, 1911	Kiefer, Okla.	68.6	3.90	None	None	1.30	2.66	2.19	3.85	1.21	1.070	5.0
2478	Mar. 1, 1913	Charleston, W. Va.	22.0	None	None	None	0.74	2.24	1.29	2.54	1.73	0.427	0.5
A-1-5	Feb. 10, 1910	Grove City, Pa.	15.0	None	None	None	0.63	2.04	1.02	2.06	2.00	0.315	Dry

(a) Not determined.

QUESTIONNAIRE FOR NATURAL GAS SURVEY

Date....., Well or farm name....., No.....

Location, twp....., R....., sec....., quarter....., (or lot).....

Location on sec. or lot, ft. from.....N.....E.....S.....W, line.

Owner..... Address.....

Lessee..... Address.....

Driller..... Address.....

Record of sands:	*ft. to top.*	*ft. thick.*	*Character of sand.*
1st or			
2d or			
3d or			
4th or			

Total depth of well.....ft.; diameter....., curbing.....

Date when well was completed.....month,.....Year

Days flowing before being capped..... Approx. volume.....cu. ft.,

Approximate gas well has delivered to date.....cu. ft.,

Open flow: Initial.....cu. ft.; last test.....date;.....cu. ft.,

Rock pressure: Initial.....lbs.; last test.....date;.....lbs.

(Indicate whether gaged or estimated.)

Character of gas (dry, gas and oil, casinghead, etc.).....

Present condition of well.....

Location, distance and direction of other wells within 100 yds.....

.....

Additional information.....

.....

.....

.....

.....

(Should include data if gasoline is extracted, etc.)

Data furnished by..... of ..... Company,

(If additional space is required use back of this sheet.)

Laboratory number.....,

Sampled by..... Date.....

Sample received..... Analyzed.....

Analysis:	CO_2	HHC	O_2	CO	H_2	CH_4	C_2H_6	Res.	B.t.u.
Per Cent.									

Remarks.....

.....

.....

.....

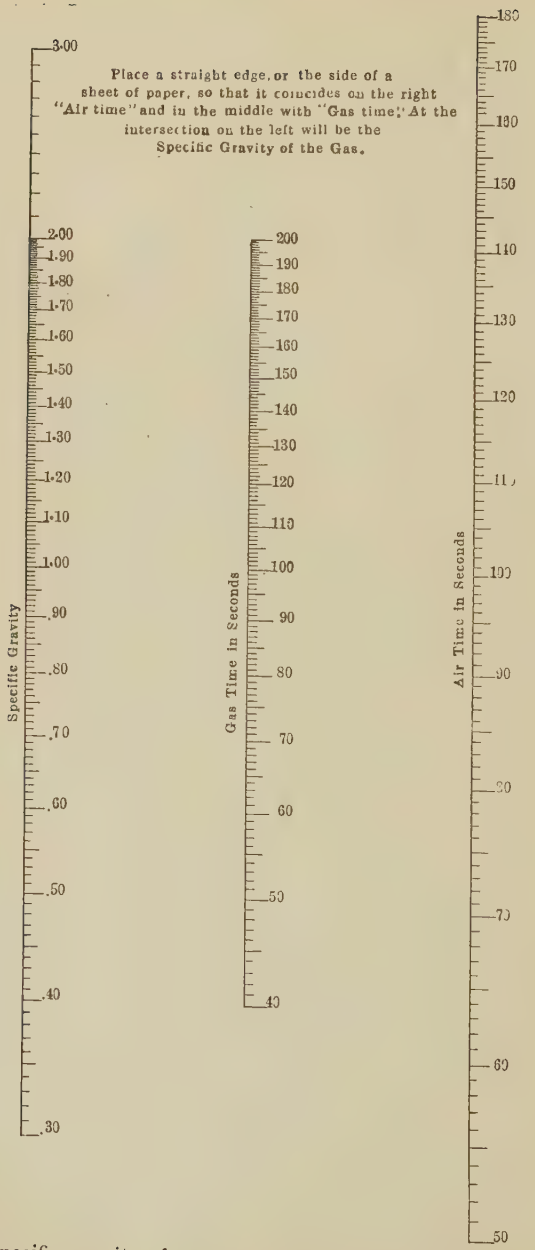


FIG 139.—Specific gravity chart for natural gas. (Computed by Frank P. Peterson & Co.)

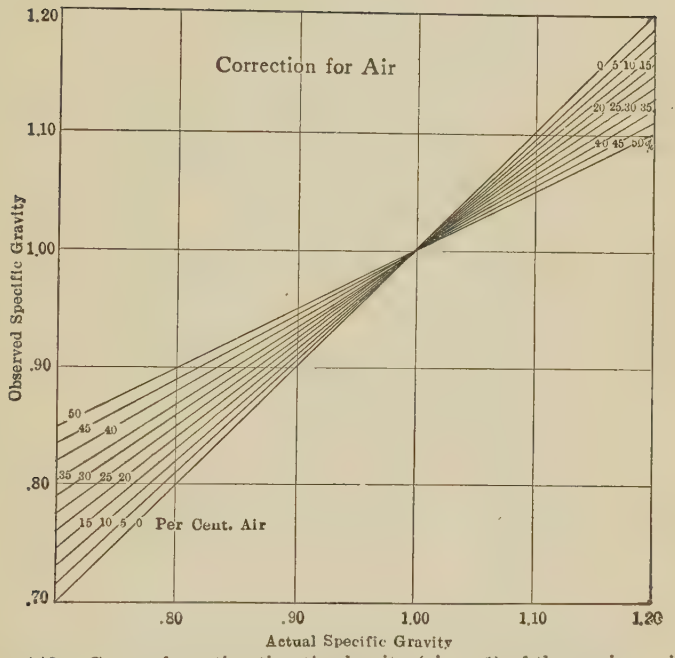


FIG. 140.—Curves for estimating the density (air = 1) of the gas in a mixture of gas and air. (From Westcott's "Handbook of Casing Head Gas." These curves are only approximately correct, but serve for practical purposes.

The Gasoline Yield of Gases of Various Specific Gravities Determined by Either the Edwards Gas Balance Method or the Effusion Method, According to Westcott

Specific gravity (air = 1)	Gallons of gasoline per 1000 cu. ft.
0.57	0.00
0.58	0.00
0.65	0.12
0.67	0.23
0.68	0.39
0.75	0.50
0.77	0.31
0.77	0.34
0.78	0.90
0 80	1.00
0.83	1.00
0.85	0.85
0.86	1.13
0.87	0.69
0.90	1.75
0.90	2.00
0.93	0.80
0.93	1.09
0.99	1 21
1.00	2.00
1.02	1.15
1.02	1.24
1.03	2.50
1.07	1.12
1.03	1.13
1.03	1.22
1.07	0.82
1.01	1.05
1.07	3.00
1.09	1.55
1.07	0.92
1.07	0.98
1.03	1.26
1.03	1.22
1.00	2.50
1.21	3.50
1.29	2.12
1.33	2.80
1.29	3.00
1.37	3.50
1.38	4.00
1.46	5.00

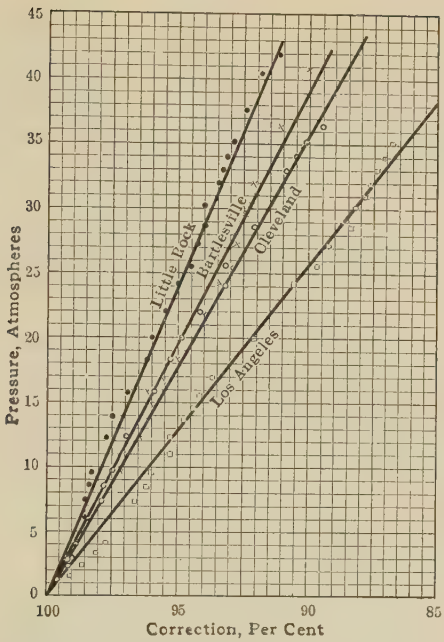


FIG. 141.—Compressibility curves of natural gas from Little Rock, Ark., Bartlesville, Okla., Cleveland, Ohio, and Los Angeles, Cal. (Bureau of Mines *Tech. Paper* **158.**)

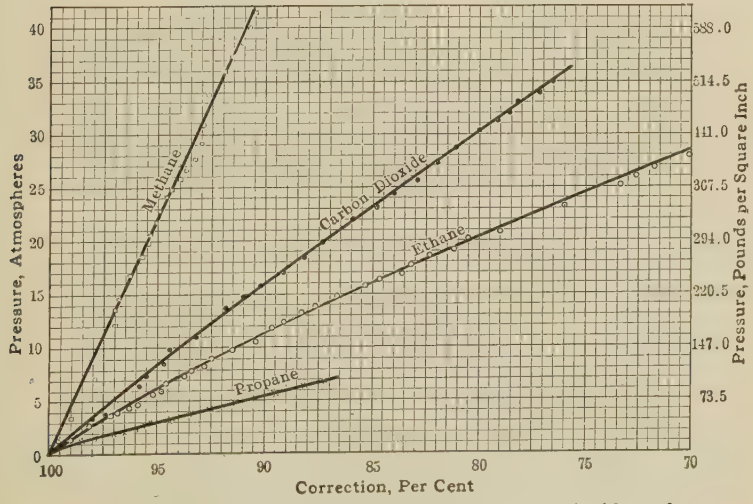


FIG. 142.—Compressibility curves of methane, carbon dioxide, ethaae, and propane. (From Bureau of Mines *Tech. Paper* **158.**)

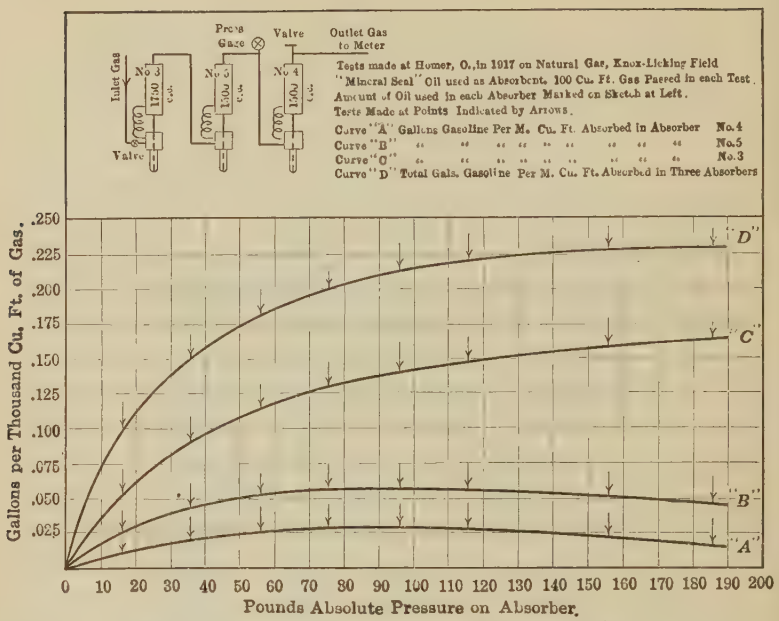

Press Gage
Valve
Outlet Gas to Meter
Inlet Gas
No. 3
No. 5
No. 4
1750 c.c.
1500 c.c.
1500 c.c.
Valve
Tests made at Homer, O., in 1917 on Natural Gas, Knox-Licking Field
"Mineral Seal" Oil used as Absorbent. 100 Cu. Ft. Gas Passed in each Test.
Amount of Oil used in each Absorber Marked on Sketch at Left.
Tests Made at Points Indicated by Arrows.
Curve "A" Gallons Gasoline Per M. Cu. Ft. Absorbed in Absorber No. 4
Curve "B" " " " " " " " " " No. 5
Curve "C" " " " " " " " " " No. 3
Curve "D" Total Gals. Gasoline Per M. Cu. Ft. Absorbed in Three Absorbers
Gallons per Thousand Cu. Ft. of Gas.
.250
.225
.200
.175
.150
.125
.100
.075
.050
.025
"D"
"C"
"B"
"A"
0 10 20 30 40 50 60 70 80 90 100 110 120 130 140 150 160 170 180 190 200
Pounds Absolute Pressure on Absorber.

FIG. 143.—The effect of pressure upon absorption.

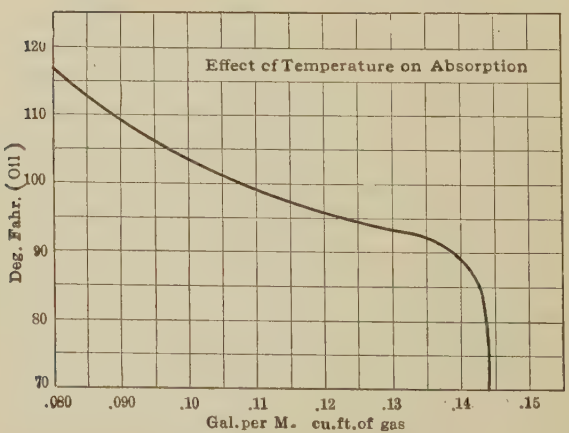


FIG. 144.—The effect of temperature on absorption. (R. O. Neal, Bureau of Mines.)

A Select Bibliography of the Extraction of Gasoline from Natural Gas

1912—Burrell, George A., and Allen, Irving C. Liquefied products from natural gas, their properties and uses. 1912. (*Tech. Paper* **10,** Bureau of Mines.)

Deals with the development of gas liquefaction by pressure, its early attempts, later improvements, and experiments in liquefying crude natural gas. Efficiency of liquefied product for definite uses. Handling of natural gas products other than liquefied gas.

1913—Burrell, George A., and Seibert, Frank M. The sampling and examination of mine gases and natural gas. 1913. (*Bulletin* **42,** Bureau of Mines.)

Tests for gasoline content of natural gas. Natural gas a source of gasoline. Principles involved in the manufacture of gasoline from natural gas.

1915—Matthews, F. E. Gasoline from natural gas. 1915. (*Power,* **42,** 677 and 678.)

The process described consists of compressing the gas and then cooling it. A table is given of the chemical constituents and commercial products derived from crude petroleum and natural gas.

1916—Burrell, G. A., and Jones, G. W. Methods of testing natural gas for gasoline content. 1916. (*Tech. Paper* **87,** Bureau of Mines.)

Natural gas to be treated by compression and condensation methods may be tested for its gasoline content by determining the specific gravity of the gas and the percentage of absorption in oil.

Burrell, G. A., Biddison, P. M., Oberfell, G. G. The extraction of gasoline from natural gas by absorption methods. 1916. (*Met. Chem. Eng.,* **14,** 651–660.)

The article deals with a method of extracting gasoline from natural gas by absorbing the gasoline in oil and subsequently separating the gasoline from the oil by distillation.

1916—Gasoline from natural gas. 1916. (*Sci. Am. Suppl.,* **81,** 187.)

A general review of the history and development of the industry.

Glick, Bernard N. Gasoline from natural gas. 1916. (*Automobile,* **35,** 777–80.)

Three processes are discussed: compression and condensation, refrigeration and absorption processes.

1917—Burrell, George A., Biddison, P. M., and Oberfell, G. G. Extraction of gasoline from natural gas by absorption methods. 1917. (*Bulletin* **120,** Bureau of Mines.)

Describes the manufacture of gasoline from casinghead gas. Development of absorption process. Occurrence of gasoline in natural gas. Analysis and tests of natural gas used in experiment.

1918—Dykema, W. P. Recovery of gasoline from natural gas by compression. 1918. (*Bulletin* **151,** Bureau of Mines.) Reprinted in *Petroleum Rev.,* **39,** Nos. 841–56, Aug.–Dec., 1918.

General description of the process followed and mention made of the history of the industry, factors to be considered in examining field from which the gas is to be taken, methods of collecting natural gas, data on plant practice, condensate, the use of ammonia as an auxiliary cooling agent, machines used in compression plants, costs of compression plants, etc.

Oberfell, G. G. Testing natural gas for gasoline. 1918. (*J. Ind. Eng. Chem.*, **10,** 211–14.)

Deals with casinghead gas and describes the method of operating and of distillation. A table is attached giving a summary of the results obtained with the cubic-foot absorber.

1919—Bendit, Louis. Casinghead gasoline process discussed. 1919. (*Oil & Gas J.*, **17,** 62–63.) Reprinted in *Petroleum Times*, **2,** 15–17.

Description of the various methods of testing gas and the extraction of gasoline therefrom. Wide variation in yield of different gases in many fields.

Burrell, G. A. Natural gas gasoline plants. 1919. (*Nat. Gas & Gasoline*, **13,** 57–61; also in *Gas Record*, **15,** 105–108; *Petroleum Age*, **6,** 101–4.)

The article was written with a view of bringing out errors that are being made in plant operations or mistakes in plant design.

1919—Burrell, G. A., Biddison, P. M., and Oberfell, G. G. Testing natural gas for gasoline content. 1919. (*Water & Gas Rev.*, **29,** 12–13.)

Deals with the laboratory method, the analyses and tests of natural gas used in experiments and tests with small portable absorber. Table given of results of tests of fresh and treated natural gas.

Burrell, Geo. A., Biddison, P. M., and Oberfell, G. G. Extraction of gasoline from natural gas by absorption methods. 1919. (*Water & Gas Rev.*, **29,** 25–6.)

Extract from *Bulletin* **120,** Bureau of Mines, by the same authors.

Dykema, W. P. Recent developments in the absorption process for recovering gasoline from natural gas. 1919. (*Bulletin* **176,** Bureau of Mines.)

Discusses the character of natural gas treated by absorption, plant design, types of testers and use of same. Cost of plants and records of same.

Dykema, W. P., and Neal, Roy O. Determining gasoline in natural gas. 1919. (*Automotive Ind.*, **40,** 57–59; *Chem. Eng.*, **27,** 5–6.)

Description of a new absorption method evolved at the Bartlesville Experiment Station. The method is effected by using an absorption apparatus, which is constructed according to the principle of the Friederich wash-bottle.

Northrup, John D. Gasoline from natural gas. 1919. (*Salt Lake Min. Rev.*, **21,** 27–8.)

Description of the history of the industry, the method followed and the amount of gasoline produced.

Oberfell, G. G., and Burrell, G. A. Testing gas for the gasoline content. 1919. (*Oil & Gas J.*, **18,** 70–72.)

Information by two chemical engineers on the methods followed in gasoline extraction.

Oberfell, G. G., Shinkle, S. D., and Meserve, S. B. Testing natural gas for gasoline content. 1919. (*J. Ind. Eng. Chem.*, **11,** 197–200.)

The principle of this method for testing natural gas for gasoline content consists in absorbing the vapors in a solid absorbing medium such as charcoal and subsequently recovering gasoline by distillation.

Spencer, Jr., E. A. Gasoline from natural gas. 1919. (*Gas Engine*, **21**, 188; *Nat. Gas & Gasoline*, **13**, 5.)

States that the by-products of the gasoline manufacture should be saved and advises as the best method the absorption process.

1919—Spencer, E. A. Gasoline recovery. 1919. (*Nat. Gas & Gasoline*, **13**, 51–52.)

The absorption process described and illustrated, step by step. In West Virginia, 100,000 gal. of gasoline are daily recovered from natural gas and most of it is manufactured under the Geo. A. Saybolt patent rights (U. S. Patent No. 889927, April 18, 1911), held by the Hope Natural Gas Co., of Pittsburgh, Pa. The whole process is continuous. The gas passes countercurrent to the oil and the oil absorbs from the gas the gasoline content.

Capacities, in Cu. Ft. per 24 Hours, of Thin Plate Orifices

Capacities, in Cubic Feet per Twenty-four Hours, of a One-sixteenth-Inch Thin Plate Orifice.

Thickness of Plate, One-eighth Inch.

Used in Testing Small Natural and Casinghead Gas Wells.

Specific Gravities—0.6 to 1.7.

Temperature—60°F. Atmospheric Pressure—14.4

Pressure in. of water	0.6	0.65	0.7	0.75	0.8	0.85	0.9	0.95	1.0
0.5	94	90	87	84	82	79	77	75	73
1.0	139	134	129	125	121	117	114	111	108
1.5	173	166	160	155	150	145	141	137	134
2.0	204	196	189	182	177	171	166	162	158
2.5	226	217	209	202	196	190	184	180	175
3.0	248	238	229	222	215	208	202	197	192
3.5	270	259	250	241	234	227	220	214	209
4.0	290	279	269	260	252	244	237	231	225
4.5	308	296	286	276	267	259	252	245	239
5.0	325	313	301	291	282	273	266	258	252
5.5	342	329	317	306	296	287	279	272	265
6.0	358	344	331	320	310	300	292	284	277

Pressure in. of water	1.05	1.10	1.15	1.20	1.30	1.40	1.50	1.60	1.70
0.5	71	69	68	67	64	62	60	58	56
1.0	105	103	101	99	95	91	88	85	83
1.5	131	128	125	122	117	113	109	106	103
2.0	154	151	147	144	139	134	129	125	121
2.5	171	167	163	160	153	148	143	138	134
3.0	187	183	179	175	168	162	157	152	147
3.5	204	199	195	191	183	177	171	165	160
4.0	219	214	210	205	197	190	184	178	173
4.5	233	228	223	218	210	202	195	189	183
5.0	246	240	235	230	221	213	206	199	193
5.5	258	253	247	242	232	224	216	209	203
6.0	270	264	258	253	243	234	226	219	212

Capacities, in Cubic Feet per Twenty-four Hours, of a One-eighth-inch Thin Plate Orifice

Thickness of Plate, One-eighth Inch
Used in Testing Small Natural and Casinghead Gas Wells
Specific Gravities—0.6 to 1.7

Temperature—60°F. Atmospheric Pressure—14.4

Pressure in. of water	0.6	0.65	0.7	0.75	0.8	0.85	0.9	0.95	1.0
0.5	465	446	430	416	402	390	379	369	360
1.0	650	625	602	582	563	547	531	517	504
1.5	818	786	758	732	709	688	668	650	634
2.0	938	902	869	839	813	788	766	746	727
2.5	1065	1023	986	953	922	895	870	846	825
3.0	1162	1116	1076	1039	1006	976	949	923	900
3.5	1273	1223	1178	1138	1102	1069	1039	1012	986
4.0	1356	1302	1255	1212	1174	1139	1107	1077	1050
4.5	1441	1384	1334	1289	1248	1210	1176	1145	1116
5.0	1523	1464	1410	1363	1319	1280	1244	1210	1180
5.5	1607	1544	1488	1438	1392	1350	1312	1277	1245
6.0	1678	1612	1554	1500	1453	1410	1370	1334	1300

Pressure in. of water	1.05	1.10	1.15	1.20	1.30	1.40	1.50	1.60	1.70
0.5	351	343	336	329	316	304	294	285	276
1.0	491	481	470	460	442	426	411	398	387
1.5	618	604	591	579	556	536	518	501	486
2.0	709	693	678	664	638	614	593	575	558
2.5	804	787	769	753	724	697	674	652	633
3.0	878	858	839	822	789	761	735	711	690
3.5	961	940	919	900	865	833	805	779	756
4.0	1024	1001	979	958	921	887	857	830	805
4.5	1088	1064	1041	1019	979	943	911	882	856
5.0	1151	1125	1100	1077	1035	997	963	933	905
5.5	1214	1187	1161	1136	1092	1052	1016	984	955
6.0	1268	1239	1212	1187	1140	1099	1061	1028	997

Capacities, in Cubic Feet per Twenty-four Hours, of a One-quarter-inch Thin Plate Orifice

Thickness of Plate, One-eighth Inch
Used in Testing Small Natural and Casinghead Gas Wells
Specific Gravities—0.6 to 1.7

Temperature—60°F. Atmospheric Pressure—14.4

Pressure in. of water	0.6	0.65	0.7	0.75	0.8	0.85	0.9	0.95	1.0
0.5	1181	1135	1094	1056	1023	992	965	938	915
1.0	1575	1513	1458	1409	1364	1323	1286	1252	1220
1.5	1936	1860	1792	1732	1677	1627	1581	1538	1500
2.0	2259	2170	2092	2020	1965	1898	1845	1795	1750
2.5	2582	2480	2390	2310	2236	2169	2108	2052	2000
3.0	2827	2716	2617	2529	2448	2375	2308	2247	2190
3.5	3060	2940	2833	2737	2650	2570	2498	2431	2370
4.0	3266	3138	3024	2921	2828	2744	2666	2596	2530
4.5	3474	3336	3215	3106	3007	2918	2835	2760	2690
5.0	3640	3498	3370	3256	3153	3058	2972	2893	2820
5.5	3834	3684	3550	3429	3320	3221	3130	3046	2970
6.0	4002	3845	3705	3580	3466	3362	3268	3180	3100

Pressure in. of water	1.05	1.10	1.15	1.20	1.30	1.40	1.50	1.60	1.70
0.5	893	872	853	835	802	773	747	723	702
1.0	1191	1163	1138	1114	1070	1031	996	964	936
1.5	1464	1430	1398	1369	1315	1268	1225	1186	1150
2.0	1708	1668	1632	1597	1535	1479	1428	1383	1342
2.5	1952	1906	1865	1825	1754	1690	1633	1581	1534
3.0	2137	2088	2042	2000	1921	1851	1788	1731	1680
3.5	2312	2260	2210	2163	2078	2003	1935	1873	1818
4.0	2469	2412	2359	2309	2219	2138	2065	2000	1940
4.5	2625	2565	2508	2455	2359	2273	2196	2126	2063
5.0	2752	2688	2630	2574	2473	2383	2302	2229	2163
5.5	2898	2831	2770	2711	2605	2510	2425	2348	2278
6.0	3025	2955	2890	2830	2718	2620	2531	2450	2377

Capacities, in Cubic Feet per Twenty-four Hours, of a Three-eighths-inch Thin Plate Orifice

Thickness of Plate, One-eighth Inch
Used in Testing Small Natural and Casinghead Gas Wells
Specific Gravities—0.6 to 1.7

Temperature—60°F. Atmospheric Pressure—14.4

Pressure in. of water	0.6	0.65	0.7	0.75	0.8	0.85	0.9	0.95	1.0
0.5	2270	2180	2100	2030	1970	1910	1850	1810	1760
1.0	3460	3320	3200	3090	3000	2910	2820	2750	2680
1.5	4310	4140	3990	3860	3730	3620	3520	3420	3339
2.0	4830	4640	4470	4320	4180	4060	3940	3840	3740
2.5	5400	5190	5000	4830	4680	4540	4410	4290	4186
3.0	5770	5550	5350	5170	5000	4850	4720	4590	4474
3.5	6290	6050	5830	5630	5450	5290	5140	5000	4875
4.0	6650	6390	6160	5950	5760	5590	5430	5280	5152
4.5	7210	6930	6680	6450	6240	6060	5890	5730	5585
5.0	7680	7380	7110	6870	6650	6450	6270	6100	5946
5.5	8100	7790	7500	7250	7020	6810	6620	6440	6278
6.0	8290	7970	7680	7420	7180	6970	6770	6590	6423

Pressure in. of water	1.05	1.10	1.15	1.20	1.30	1.40	1.50	1.60	1.70
0.5	1720	1680	1640	1610	1540	1490	1440	1390	1350
1.0	2620	2550	2500	2450	2350	2260	2190	2120	2050
1.5	3260	3180	3110	3050	2930	2820	2730	2640	2560
2.0	3650	3560	3490	3410	3280	3160	3050	2960	2870
2.5	4080	3990	3900	3820	3670	3540	3420	3310	3210
3.0	4370	4260	4170	4080	3920	3780	3650	3540	3430
3.5	4760	4650	4550	4450	4270	4120	3980	3850	3740
4.0	5030	4910	4800	4700	4520	4350	4210	4070	3950
4.5	5450	5330	5210	5100	4900	4720	4560	4410	4280
5.0	5800	5670	5540	5430	5210	5020	4850	4700	4560
5.5	6130	5980	5850	5730	5510	5310	5130	4960	4810
6.0	6270	6120	5990	5860	5630	5430	5240	5080	4930

Capacities, in Cubic Feet per Twenty-four Hours, of a One-half-inch Thin Plate Orifice

Thickness of Plate, One-eighth Inch
Used in Testing Small Natural and Casinghead Gas Wells
Specific Gravities—0.6 to 1.7

Temperature—60°F. Atmospheric Pressure—14.4

Pressure in. of water	0.6	0.65	0.7	0.75	0.8	0.85	0.9	0.95	1.0
0.5	4,490	4,320	4,160	4,020	3,890	3,770	3,670	3,570	3,480
1.0	6,260	6,010	5,790	5,600	5,440	5,260	5,110	4,970	4,850
1.5	7,900	7,590	7,310	7,070	6,840	6,640	6,450	6,280	6,120
2.0	9,140	8,780	8,460	8,170	7,910	7,680	7,460	7,260	7,080
2.5	10,220	9,820	9,470	9,140	8,850	8,590	8,350	8,120	7,920
3.0	11,150	10,720	10,330	9,980	9,660	9,370	9,110	8,860	8,640
3.5	12,020	11,550	11,130	10,750	10,410	10,100	9,810	9,550	9,310
4.0	12,800	12,290	11,850	11,440	11,080	10,750	10,450	10,170	9,910
4.5	13,480	12,950	12,480	12,050	11,670	11,320	11,000	10,710	10,440
5.0	14,130	13,570	13,080	12,640	12,230	11,870	11,530	11,230	10,940
5.5	14,690	14,110	13,600	13,130	12,720	12,340	11,990	11,670	11,380
6.0	15,210	14,620	14,080	13,610	13,170	12,780	12,420	12,090	11,780

Pressure in. of water	1.05	1.10	1.15	1.20	1.30	1.40	1.50	1.60	1.70
0.5	3,400	3,320	3,250	3,180	3,050	2,940	2,840	2,750	2,670
1.0	4,730	4,620	4,520	4,420	4,250	4,100	3,960	3,830	3,720
1.5	5,970	5,830	5,710	5,590	5,370	5,170	5,000	4,840	4,690
2.0	6,910	6,750	6,600	6.460	6,210	5,980	5,780	5,600	5,430
2.5	7,730	7,550	7,380	7,230	6,950	6,690	6,470	6,260	6,070
3.0	8,430	8,240	8,060	7,890	7,580	7,300	7,050	6,830	6,630
3.5	9,090	8,880	8,680	8,500	8,170	7,870	7,600	7,361	7,140
4.0	9,670	9,450	9,240	9,050	8,690	8,380	8,090	7,840	7,600
4.5	10,190	9,950	9,730	9,530	9,160	8,820	8,520	8,250	8,010
5.0	10,680	10,430	10,200	9,990	9,600	9,250	8,930	8,650	8,390
5.5	11,100	10,850	10,610	10,380	9,980	9,610	9,290	8,990	8,720
6.0	11,500	11,230	10,990	10,760	10,330	9,960	9,620	9,310	9,040

Capacities, in Cubic Feet per Twenty-four Hours, of a Three-quarter-inch Thin Plate Orifice

Thickness of Plate, One-eighth Inch
Used in Testing Small Natural and Casinghead Gas Wells
Specific Gravities—0.6 to 1.7

Temperature—60°F. Atmospheric Pressure—14.4

Pressure in. of water	0.6	0.65	0.7	0.75	0.8	0.85	0.9	0.95	1.0
0.5	10,560	10,150	9,780	9,450	9,150	8,880	8,630	8,400	8,180
1.0	14,530	13,960	13,450	13,000	12,580	12,210	11,860	11,550	11,260
1.5	17,720	17,030	16,410	15,850	15,350	14,890	14,470	14,080	13,730
2.0	20,390	19,590	18,870	18,230	17,650	17,130	16,650	16,200	15,790
2.5	22,740	21,850	21,050	20,340	19,700	19,110	18,570	18,070	17,620
3.0	24,880	23,900	23,030	22,250	21,550	20,900	20,310	19,770	19,270
3.5	26,990	25,930	24,980	24,140	23,370	22,670	22,030	21,450	20,900
4.0	28,970	27,830	26,820	25,910	25,090	24,340	23,650	23,020	22,440
4.5	30,800	29,590	28,510	27,550	26,670	25,870	25,150	24,470	23,860
5.0	32,500	31,230	30,090	29,070	28,150	27,210	26,540	25,830	25,180
5.5	34,080	32,740	31,530	30,480	29,510	28,630	27,830	27,080	26,400
6.0	35,630	34,230	32,990	31,870	30,860	29,940	29,090	28,320	27,600

Pressure in. of water	1.05	1.10	1.15	1.20	1.30	1.40	1.50	1.60	1.70
0.5	7,990	7,800	7,630	7,470	7,170	6,920	6,680	6,470	6,280
1.0	10,980	10,730	10,500	10,280	9,870	9,510	9,190	8,900	8,630
1.5	13,400	13,090	12,800	12,530	12,040	11,600	11,210	10,850	10,530
2.0	15,410	15,060	14,730	14,420	13,850	13,350	12,890	12,480	12,110
2.5	17,190	16,800	16,430	16,080	15,450	14,890	14,380	13,930	13,510
3.0	18,810	18,380	17,970	17,590	16,900	16,290	15,730	15,230	14,780
3.5	20,400	19,930	19,490	19,080	18,330	17,670	17,070	16,530	16,030
4.0	21,900	21,400	20,920	20,480	19,680	18,960	18,320	17,740	17,210
4.5	23,280	22,750	22,250	21,770	20,920	20,160	19,480	18,860	18,300
5.0	24,570	24,000	23,580	22,980	22,080	21,280	20,550	19,900	19,310
5.5	25,760	25,170	24,620	24,100	23,150	22,310	21,550	20,870	20,250
6.0	26,930	26,310	25,740	25,200	24,200	23,330	22,530	21,820	21,170

Capacities, in Cubic Feet per Twenty-four Hours, of a One-inch Thin Plate Orifice

Thickness of Plate, One-eighth Inch
Used in Testing Small Natural and Casinghead Gas Wells
Specific Gravities—0.6 to 1.7

Temperature—60°F. Atmospheric Pressure—14.4

Pressure in. of water	0.6	0.65	0.7	0.75	0.8	0.85
1	26,440	25,440	24,500	23,660	22,920	22,220
2	37,510	36,040	34,750	33,600	32,520	31,530
3	46,440	44,640	43,000	41,540	40,240	39,020
4	52,630	50,590	48,740	47,060	45,600	44,200
5	57,880	55,630	53,610	51,790	50,160	48,640
6	63,140	60,720	58,480	56,490	54,720	53,060
7	68,110	65,470	63,090	60,910	59,040	57,210
8	73,050	70,220	67,680	65,350	63,310	61,390
9	77,680	74,680	72,000	69,500	67,340	65,280
10	82,340	79,150	76,270	73,650	71,370	69,190
11	86,680	83,320	80,300	77,540	75,120	72,840
12	90,720	87,190	84,000	81,140	78,600	76,220
Mercury						
0.5	67,200	64,600	62,300	60,100	58,200	56,500
1	95,200	91,500	88,200	85,100	82,500	80,000
1.5	116,600	112,000	108,000	104,300	101,000	97,900
2	134,600	129,400	124,700	120,400	116,700	113,100
2.5	145,600	139,900	134,900	130,200	126,200	122,400
3	164,900	158,500	152,700	147,500	142,900	138,600
3.5	178,200	171,300	165,100	159,400	154,500	149,800
4	190,400	183,000	176,400	170,300	165,000	160,000
5	212,900	204,600	197,200	190,400	184,500	178,900
6	233,200	224,100	216,000	208,600	202,100	195,900
7	251,900	242,100	233,400	225,300	218,300	211,700
8	269,400	258,900	249,500	240,900	233,400	226,400
9	285,700	274,600	264,700	255,600	247,600	240,100
10	301,200	289,500	279,000	269,400	261,000	253,100
11	315,800	303,600	292,500	282,500	273,700	265,400
12	328,400	315,700	304,200	293,800	284,600	276,000

Capacities, in Cubic Feet per Twenty-four Hours, of a One-inch Thin Plate Orifice

Thickness of Plate, One-eighth Inch
Used in Testing Small Natural and Casinghead Gas Wells
Specific Gravities—0.6 to 1.7

Temperature—60°F. Atmospheric Pressure—14.4

Pressure in. of water	0.9	0.95	1.0	1.05	1.1	1.15
1	21,600	21,020	20,520	20,010	19,560	19,120
2	30,640	29,800	29,080	28,360	27,720	27,120
3	37,940	36,880	36,000	35,130	34,320	33,550
4	42,980	41,800	40,800	39,790	38,880	38,040
5	47,280	45,980	44,880	43,770	42,760	41,830
6	51,600	50,180	48,960	47,760	46,650	45,640
7	55,630	54,120	52,800	51,500	50,320	49,220
8	59,680	58,050	56,640	55,240	54,000	52,800
9	63,480	61,720	60,240	58,800	57,430	56,160
10	67,270	65,420	63,840	62,280	60,860	59,520
11	70,800	68,880	67,200	65,560	64,080	62,660
12	74,110	72,000	70,320	68,610	67,030	65,560
Mercury						
0.5	54,900	53,400	52,100	50,800	49,600	48,600
1	77,800	75,600	73,800	72,800	70,300	68,800
1.5	95,300	92,600	90,400	88,200	86,200	84,300
2	110,000	107,000	104,400	101,800	99,500	97,300
2.5	118,900	115,700	112,900	110,100	107,600	105,300
3	134,700	131,000	127,800	124,700	121,800	119,200
3.5	145,600	141,600	138,200	134,800	131,700	128,800
4	155,600	151,300	147,600	144,000	140,700	137,600
5	174,000	169,200	165,000	161,000	157,300	153,900
6	190,500	185,300	180,800	176,400	172,300	168,600
7	205,800	200,200	195,300	190,600	186,200	182,100
8	220,100	214,000	208,800	203,700	199,100	194,700
9	233,500	227,000	221,500	216,100	211,200	206,500
10	246,100	239,300	233,500	227,800	222,600	217,700
11	258,000	250,900	244,800	238,900	233,400	228,300
12	268,400	261,000	254,600	248,400	242,700	237,400

Capacities, in Cubic Feet per Twenty-four Hours, of a One-inch Thin Plate Orifice

Thickness of Plate, One-eighth Inch
Used in Testing Small Natural and Casinghead Gas Wells
Specific Gravities—0.6 to 1.7

Temperature—60°F. Atmospheric Pressure—14.4

Pressure in. of water	1.2	1.3	1.4	1.5	1.6	1.7
1	18,720	18,000	17,320	16,750	16,200	15,720
2	26,540	25,480	24,570	23,760	22,990	22,290
3	32,850	31,560	30,400	29,370	28,440	27,600
4	37,220	35,760	34,460	33,310	32,230	31,270
5	40,940	39,360	37,920	36,620	35,470	34,410
6	44,680	42,960	41,370	39,960	38,680	37,530
7	48,190	46,320	44,610	43,100	41,730	40,480
8	51,690	49,680	47,850	46,220	44,760	43,410
9	54,960	52,800	50,880	49,170	47,610	46,200
10	58,240	55,960	53,920	52,100	50,440	48,960
11	61,320	58,920	56,780	54,860	53,110	51,520
12	64,170	61,680	59,400	57,400	55,580	53,920
Mercury						
0.5	47,500	45,700	44,000	42,500	41,100	39,900
1	67,300	64,700	62,300	60,200	58,300	56,600
1.5	82,500	79,200	76,300	73,800	71,400	69,300
2	95,300	91,500	88,200	85,200	82,500	80,000
2.5	103,000	99,000	95,400	92,200	89,200	86,500
3	116,600	112,000	108,000	104,300	101,000	98,000
3.5	126,100	121,200	116,700	112,800	109,200	105,900
4	134,700	129,400	124,700	120,500	116,600	113,200
5	150,600	144,700	139,400	134,700	130,400	126,500
6	165,000	158,500	152,700	147,600	142,900	138,600
7	178,200	171,200	165,000	159,400	154,300	149,700
8	190,600	183,100	176,400	170,500	165,000	160,100
9	202,100	194,200	187,100	180,800	175,000	169,800
10	213,100	204,700	197,300	190,600	184,500	179,000
11	223,400	214,700	206,800	199,900	193,500	187,700
12	232,400	223,300	215,100	207,900	201,200	195,200

Capacities, in Cubic Feet per Twenty-four Hours, of a One-and-one-quarter-inch Thin Plate Orifice

Thickness of Plate, One-eighth Inch
Used in Testing Small Natural and Casinghead Gas Wells
Specific Gravities—0.6 to 1.7
Temperature—60°F. Atmospheric Pressure—14.4

Pressure in. of water	0.6	0.65	0.7	0.75	0.8	0.85	0.9	0.95	1.0
0.5	32,780	31,490	30,350	29,320	28,390	27,540	26,760	26,050	25,390
1.0	46,260	44,440	42,830	41,380	40,060	38,860	37,770	36,760	35,830
1.5	56,600	54,380	52,410	50,630	49,020	47,560	46,210	44,980	43,850
2.0	64,840	62,300	60,040	58,000	56,160	54,480	52,940	51,530	50,230
2.5	72,400	69,570	67,040	64,760	62,710	60,830	59,120	57,540	56,090
3.0	77,980	74,920	72,200	69,750	67,540	65,520	63,670	61,970	60,410
3.5	84,490	81,180	78,220	75,570	73,170	70,980	68,980	67,140	65,450
4.0	91,400	87,810	84,620	81,750	79,150	76,790	74,620	72,630	70,800
4.5	97,810	93,980	90,560	87,490	84,710	82,180	79,860	77,730	75,770
5.0	103,260	99,210	95,610	92,370	89,430	86,760	84,310	82,060	80,000
5.5	107,230	103,020	99,280	95,910	92,870	90,090	87,550	85,210	83,060

Pressure in. of water	1.05	1.10	1.15	1.20	1.30	1.40	1.50	1.60	1.70
0.5	24,779	24,210	23,680	23,180	22,270	21,458	20,730	20,070	19,470
1.0	34,968	34,160	33,410	32,710	31,420	30,280	29,250	28,320	27,480
1.5	42,790	41,810	40,890	40,030	38,460	37,060	35,800	34,670	33,630
2.0	49,020	47,890	46,840	45,850	44,050	42,450	41,010	39,710	38,520
2.5	54,735	53,480	52,300	51,200	49,190	47,400	45,800	44,340	43,020
3.0	58,950	57,600	56,330	55,150	52,980	51,050	49,320	47,760	46,330
3.5	63,870	62,400	61,030	59,750	57,400	55,310	53,440	51,740	50,200
4.0	69,090	67,500	66,020	64,630	62,090	59,840	57,810	55,970	54,300
4.5	73,940	72,240	70,650	69,170	66,450	64,040	61,860	59,900	58,110
5.0	78,070	76,280	74,600	73,030	70,160	67,610	65,320	63,240	61,360
5.5	81,060	79,194	77,450	75,820	72,850	70,200	67,820	65,660	63,700

Methods of Testing Lubricants, Adopted by the Committee on Standardization of Petroleum Specifications (1920).

FLASH-POINT

This test shall be made in the Cleveland Open-Cup Tester, the apparatus being used without any bath or outer cup surrounding the oil cup. The oil cup should have two marks on the inside: the first, ¼ in. below the top; and the second, ⅜ in. below, the first to be used when testing oils with a flash-point below 425°F. and the second when testing oils with a flash-point at or above 425°F. The clean oil cup should be inserted into the tripod ring, which must be level, and the cup filled to the proper mark with the oil to be tested. Care should be exercised not to spill any oil on the sides or top of the cup; and if this accident should happen, all such oil must be carefully removed. A "bulb immersion" thermometer should then be inserted into the oil and suspended from a suitable support. The bulb of the thermometer should be ⅜ to ⅝ in. in length. During the test the bulb must be fully

covered by the oil and the bottom of the thermometer must not be less than ¼ in. from the bottom of the cup. The thermometer must be suspended in the oil midway between the center and inside edge of the cup. The alcohol or gas burner is then placed under the oil cup so as to heat it uniformly. The oil may be heated rapidly at first, but the rate of heating should be 8 to 10°F. (5°C.) per minute during the last 80 degrees of heating prior to attaining the flash-point. As the flash-point is approached, a test is made for every 5°F. rise in temperature (on the readings which are multiples of 5) by slowly passing a small bead-like test flame, not exceeding ⅛ in. in length, across the center of the cup ¼ in. above the surface of the oil, the movement occupying one second.

The temperature when a flame first jumps from the test flame to the oil is called the flash-point of the oil. The test must be made where the cup is free from draughts and must also be made in a subdued light.

FIRE-POINT

After the flash-point has been obtained, the same method of testing shall be continued until the oil takes fire and continues to burn. The temperature at which the oil continues to burn is the fire-point of the oil.

VISCOSITY

(A. S. T. M. Method D47–19T)

Viscosity shall be determined by means of the Saybolt Standard Universal Viscosimeter as described in the *Proceedings of the American Society for Testing Materials*, **19** (1919), i, 728.

Viscosity shall be determined at 100°F. (37.8°C.), 130°F. (54.4°C.), or 210°F. (98.9°C.). The bath shall be held constant within 0.25°F. (0.14°C.), at such a temperature as will maintain the desired temperature in the standard oil tube. For viscosity determinations at 100 and 130°F., oil or water may be used as the bath liquid. For viscosity determinations at 210°F., oil shall be used as the bath liquid. The oil for the bath liquid should be a pale engine oil of at least 350°F. flash-point (open-cup). Viscosity determinations shall be made in a room free from draughts and from rapid changes in temperature. All oil introduced into the standard oil tube, either for cleaning or for test, shall first be passed through the strainer.

To make the test, heat the oil to the necessary temperature and clean out the standard oil tube with the plunger, using some of the oil to be tested. Place the cork stopper into the lower end of the air chamber at the bottom of the standard oil tube. The stopper should be sufficiently inserted to prevent the escape of air, but should not touch the small outlet tube of the standard oil tube. Heat the oil to be tested, outside the viscosimeter, to slightly below the temperature at which the viscosity is to be determined, and pour it into the standard oil tube until it ceases to overflow into the overflow cup. By means of the oil tube thermometer keep the oil in the standard oil tube well stirred and also stir well the oil in the bath. It is extremely important that the temperature of the oil in the oil-bath be maintained constant during the entire time consumed in making the test. When the temperature of the oil in the bath and in the standard oil tube are constant and the oil in the standard oil tube is at the desired temperature, withdraw the oil tube thermometer; quickly remove the surplus oil from the overflow cup by means of a pipette so that the level of the oil in the overflow cup is below the level of the oil in the tube proper; place the 60-c.c. flask in position so that the oil from the outlet tube will flow into the flask without making bubbles; snap the cork from its position and at the same instant start the stop watch. Stir the liquid in the bath during the run and carefully maintain it at the previously determined proper temperature. Stop the watch when the bottom of the meniscus of the oil reaches the mark on the neck of the receiving flask.

The time in seconds for the delivery of 60 c.c. of oil is the Saybolt viscosity of the oil at the temperature at which the test was made. (On this determination, see also page 563.)

POUR TEST

(A. S. T. M. Method D47–18)

1. The pour test indicates the temperature at which a sample of oil in a cylindrical container of specified diameter and length will just flow under specified conditions.

2. Apparatus: The apparatus for the pour test consists of the following:

(*a*) Glass jar, approximately 1¼ in. inside diameter and 4 to 5 in. high, provided with a tightly fitting cork.

(*b*) Mercury thermometer, fitted securely in the cork so that the shaft will be held centrally in the jar with the tip of the bulb ½ in. from the bottom. The thermometer specially made for this test has a bulb ¼ to ⅜ in. long.

(*c*) Metal jacket, closely fitted around the glass jar and provided at the bottom with a disk of cork felt about ¼ in. thick.

3. Method: Place the oil in the jar to a depth of about 1¼ in. or to a sufficient depth to reach ¼ in. above the bulb of the thermometer; fit the cork tightly into the jar and place the jar in the metal jacket; then place the jacket in a freezing mixture. At each drop in temperature of 5°F. (on the readings which are multiples of 5 degrees), remove the jar from the jacket and tilt it just enough to make the oil flow. The pour test of the oil shall be taken as 5 degrees higher than the reading of the thermometer when the oil has cooled so that it will not flow when the jar is tipped to a horizontal position.

4. The rate of cooling should be such that the pour test be completed in about one-half hour.

5. The materials used in the freezing mixture vary with the temperature required to cause the lubricant to solidify. Cracked ice will be sufficient for a temperature above plus 35°F. For temperatures between +15°F. and +35°F., a mixture consisting of one volume of salt and twenty volumes of ice may be used. The salt for this purpose should be very dry and fine enough to pass a 20-mesh screen. From +15° to −5°F., ice and salt in the proportions of one to two are suitable. From 0° to −25°F., a mixture of ice and calcium chloride is used. For temperatures lower than −5°F., a mixture of solid carbon dioxide and acetone is more convenient and will produce temperatures of −70°F. or less.

6. To obtain the solid carbon dioxide, invert an ordinary liquefied carbon dioxide cylinder, open the valve carefully and let the gas flow into a chamois skin bag. Rapid evaporation will cause the carbon dioxide to solidify.

7. The carbon dioxide-acetone mixture may be made as follows:

Place a sufficient amount of dry acetone in a covered copper or nickel beaker; place the beaker in an ice-salt mixture, and, when the acetone reaches +10°F. or less, add solid carbon dioxide gradually until the desired temperature is reached.

COLD TEST FOR STEAM CYLINDER AND BLACK OILS

(A. S. T. M. Method D47–18)

The object of the cold test is to determine the lowest temperature at which oil will flow from one end of a container to the other, in case it should become frozen, and the resulting solid oil stirred till it has assumed a sufficiently pasty consistency to flow. The test is conducted by freezing an ounce of the oil solid in an ordinary 4-oz. sample bottle, using a freezing mixture if necessary. A thermometer should then be introduced into the frozen mass, and, after it has become cold, the bottle containing the congealed oil is removed from the cooling medium. The frozen oil is thoroughly stirred with a thermometer until the mass will run from one end of the bottle to the other, and at this moment the temperature as indicated is recorded. The reading is the cold test of the oil.

If the figures indicating the cold test are inside the bottle and covered by the softened oil, the reading can be obtained by grasping the bottle by the neck with one hand, having in the same hand a piece of waste, which incloses the thermometer. The thermometer is then withdrawn through the waste with the other hand for a sufficient distance to enable the operator to see the end of the mercury column and read the temperature.

ACIDITY

(A. S. T. M. Method D47–18)

Accurately weigh 10 grams of the oil into a flask, add 50 c.c. of 95 per cent. alcohol which has been neutralized with weak caustic soda, and heat to the boiling-point. Agitate the flask thoroughly in order to dissolve the free fatty acids as completely as possible. Titrate while hot with aqueous tenth-normal alkali, free from carbonate, using phenolphthalein, alkali blue or turmeric as an indicator, agitating thoroughly after each addition of alkali.

To express result as percentage of oleic acid, use the following equation:

One c.c. of tenth-normal alkali = 0.0282 gram of oleic acid. Alkali, 1 c.c. of which is equivalent to 0.5 per cent. of oleic acid, may be used. (A. S. T. M. Method, "1918 Standards," 620.)

FATTY OIL

Solutions required:

(*a*) Approximately half-normal alcoholic potassium hydroxide. Dissolve 33 grams of potassium hydroxide sticks (or an equivalent amount of sodium hydroxide sticks) in 1000 c.c. of purified 92–95 per cent. ethyl alcohol. Allow to settle and filter.

(*b*) Purified Benzene. This may be prepared as follows: To 1000 c.c. of "90 per cent. benzol" add a stick of sodium hydroxide, boil for an hour, using a condenser loop inside the neck of the flask. Transfer to a large separatory funnel and add sufficient water to cause the liquid to separate into two zones. Draw off the lower zone and discard. Wash the benzene with water once. Transfer the washed benzene to an Engler distillation flask and distil up to 82°C., discarding the residue.

(*c*) Standard solution of half-normal hydrochloric acid.

(*d*) Phenolphthalein indicator. Dissolve one gram of phenolphthalein in 100 c.c. of 95 per cent. ethyl alcohol.

(*e*) Neutral gasoline.

Saponification:

Weigh 10 grams of oil into a 350-c.c. Erlenmeyer flask. Add from a pipette 50 c.c. of the alcoholic potassium hydroxide solution, followed by 25 c.c. of the purified benzene (C_6H_6). Connect with a condenser loop. Boil on steam-bath or electric hot-plate for 90 minutes, shaking occasionally. Remove and add 25 c.c. of neutral gasoline, and titrate with the half-normal hydrochloric acid solution, after adding 2 or 3 drops of the phenolphthalein indicator solution until the pink color is destroyed. The absence of the pink color may be determined after the titration has begun, by allowing the solution to stand at rest, approximately a minute, and noting the color of the lower zone. Run two blanks with the same mixture of alcoholic potassium hydroxide solution and purified benzene. From the difference between the number of cubic centimeters of half-normal acid required for the blanks and for the determination, the percentage of fatty oil may be calculated as follows:

$$\frac{\text{No. of c.c. N/2 acid used} \times 0.02805 \times 100}{0.195 \times \text{weight of oil taken}} = \text{per cent. of fatty oil.}$$

CARBON RESIDUE

(A. S. T. M. Method D47–18)

Apparatus:

(*a*) Porcelain crucible, wide form, glazed throughout, 25 to 26 c.c. capacity, 46 mm. in diameter.

(*b*) Skidmore iron crucible, 45 c.c. (1½ oz.) capacity, 65 mm. in diameter, 37 to 39 mm. high with cover, without delivery tubes and one opening closed.

(*c*) Wrought-iron crucible with cover, about 180 c.c. capacity, 80 mm. diameter, 58 to 60 mm. high. At the bottom of this crucible a layer of sand is placed about 10 mm. deep or enough to bring the Skidmore crucible with cover on nearly to the top of the wrought iron crucible.

(*d*) Triangle, pipe-stem covered, projection on side so as to allow flame to reach the crucible on all sides.

(*e*) Sheet-iron or asbestos hood, provided with a chimney about 2 to 2½ in. high, 2⅛ in. in diameter, to distribute the heat uniformly during the process.

(*f*) Asbestos, or hollow sheet-iron block, 6 to 7 in. square, 1¼ to 1½ in. high, provided with opening in center 3¼ in. in diameter at the bottom and 3½ in. in diameter at the top.

Method: The test shall be conducted as follows:

Ten grams of the oil to be tested are weighed in the porcelain crucible (*a*) which is placed in the Skidmore crucible (*b*), and these two crucibles set in the larger iron crucible (*c*), being careful to have the Skidmore crucible set in the center of the iron crucible, covers being applied to the Skidmore and iron crucibles. Place on triangle and suitable stand with asbestos block and cover with sheet-iron or asbestos hood, in order to distribute the heat uniformly during the process.

Heat from a bunsen burner or other burner is applied with a high flame surrounding

the large crucible (c) until vapors from the oil start to ignite over the crucible, when the heat is slowed down so that the vapor (flame) will come off at a uniform rate. The flame from the ignited vapors should not extend over 2 in. above the sheet-iron hood. After the vapor ceases to come off, the heat is increased as at the start and kept so for five minutes, making the lower part of large crucible red hot, after which the apparatus is allowed to cool somewhat before uncovering the crucible. The porcelain crucible is removed, cooled in a desiccator and weighed.

The entire process should require one-half hour to complete when heat is properly regulated. The time will depend somewhat upon the kind of oil tested, as a very thin, rather low flash-point oil will not take as long as a heavy, thick, high flash-point oil.

EMULSIFYING PROPERTIES

ESSENTIAL FEATURES OF EMULSIFIER

The oil and water to be emulsified are contained in an ordinary commercial 100 c.c. graduated cylinder, $1\frac{1}{16}$ to $1\frac{3}{16}$ in. inside diameter. An oil or water-bath is provided for maintaining the contents of the cylinder at a temperature of 130°F., except when a different temperature is specified, both during the stirring and subsequent settling-out of the oil from the emulsion. The paddle used in stirring is a copper plate, $4\frac{3}{4}$ in. long, between $\frac{3}{4}$ and $\frac{7}{8}$ in. wide, and $\frac{1}{16}$ in. thick. Means are provided for revolving this paddle about a vertical axis parallel to and midway between its two longer edges, and for keeping the speed fairly constant at 1500 r.p.m. Some form of holder for the cylinders is a convenience but not a necessity, since, on account of the ample clearance between paddle and cylinder, and the fact that a sample is stirred for only 5 minutes, a cylinder may be held by hand during the stirring. A stop should be provided, so that, when the paddle is lowered into the cylinder (or bath raised), the distance from the bottom of the paddle to the bottom of the cylinder will be about $\frac{1}{4}$ in. To save time otherwise lost in waiting for the filled cylinders to come to the temperature of the bath, it is desirable that the bath should be large enough to contain several cylinders.

EMULSION TEST

Forty c.c. of the emulsifying liquid are placed in a clean 100-c.c. graduated cylinder and 40 c.c. of the oil to be tested are added. The cylinder is then placed in the bath, and, when the contents have reached the temperature required for the test, they are stirred by the paddle for 5 minutes. The paddle is stopped, withdrawn from the cylinder, and wiped clean. The cylinder is then allowed to stand for the specified time and is then inspected.

DEMULSIBILITY TEST

Pour 27 c.c. of the oil to be tested and 53 c.c. of distilled water into a cylinder, place cylinder in bath and heat to 130°F. Submerge the paddle and run it for 5 minutes at a speed of 1500 r.p.m. Stop the paddle, withdraw it from the cylinder, and use the finger to wipe off the emulsion clinging to the paddle and to return it to the cylinder. Wipe off the paddle with paper, so that it will not contaminate the next sample. Keep the temperature of the cylinder constant at 130°F. and take readings every minute of the position of the line of demarcation between the topmost layer of oil and the adjoining emulsion. The first reading is taken one minute after stopping the paddle. With oils which act normally, the rate of settling out of the oil increases up to a maximum and then decreases, and the maximum value, in c.c. per hour, is called the "demulsibility" and is recorded as the numerical result of the test. Each rate of settling is the average rate calculated from the time of stopping the paddle to the time of reading, as shown in the following condensed table.

Time	Time since stopping paddle	Reading at interface between oil and emulsion	C.c. of oil settled out	Rate of settling, c.c. per hour
9.50	0	80	0	0
9.55	5	77	3	36
10.02	12	67	13	65
10.05	15	63	17	68
10.10	20	61	19	57

The demulsibility in this case would be 68, the highest value in the last column. In cases where the maximum rate of settling has not been reached at the end of one hour, the test is discontinued and the demulsibility taken as the number of c.c. which settled out in the hour.

CORROSION

A clean strip of pure copper, about ½ in. wide and 2 in. long, is heated to redness in a bunsen flame and, while red hot, dropped into alcohol. The strip is then allowed to dry as quickly as possible in the air and dropped into a sample of the oil contained in a test-tube. About half the length of the copper strip should be submerged. The test-tube is then closed with a stopper and left to stand for 24 hours. At the end of this time, the copper strip is removed and washed clean with proper solvents. It is then compared with a similar strip freshly cleaned as previously described. No discoloration of the test strip should be shown by this comparison.

EVAPORATION

Twenty grams of the oil are placed in a weighed flat-bottomed glass crystallizing dish having a diameter of approximately 3¾ in. The dish is then placed in an oven at a temperature of 212°F. for two hours, cooled in a desiccator, and weighed.

PRECIPITATION

Five c.c. of the oil are mixed with 95 c.c. of petroleum ether in a tall stoppered graduated cylinder and allowed to stand. The petroleum ether must be freshly redistilled and the portion boiling above 150°F. discarded. It must not show perceptible solubility in concentrated sulphuric acid.

PROTECTION

A clean polished steel plate, 2 in. long, ½ in. wide, and ⅛ in. thick, is coated by immersing the lubricant which has been heated to a temperature of 212°F. The plate is removed while still hot, allowed to cool in a vertical position, and suspended vertically within a 10 per cent. salt solution.

WICK-FEED TEST

An oil container made of brass of capacity of about one quart is fitted in the center with a brass tube of ½ in. internal diameter, which serves as an oil-way and which feeds into a graduated glass cylinder, where the quantity of oil fed by eight strands of worsted zephyr is measured. At the beginning of the test the wick should be dipped in the oil and the lift of the wick should be maintained at from ½ to ¼ in. The wick should be supported by a copper wire bent in a hook which grips the outlet end of the wick below the level of the oil, as is the usual manner in a wick feed.

The worsted zephyr shall be of the best quality, pure long fibre, cream white fine wool, thoroughly washed, scoured and carded. It shall be in its natural condition; not dyed nor subjected to any chemical process. Strands shall be four-ply soft spun and twisted. The separate plies shall be of uniform thickness throughout their entire length.

VISCOSITY

[A. S. T. M. Method. (Revised in 1920)]

(Received after the Methods of Testing Lubricants were adopted by the Committee)

1. Viscosity shall be determined by means of the Saybolt Standard Universal Viscosimeter.

2. (*a*) The Saybolt Standard Universal Viscosimeter is made entirely of metal. The standard oil tube *A* (see Fig. 145) is fitted at the top with an overflow cup *B*, and the tube is surrounded by a bath. At the bottom of the standard oil tube is a small outlet tube through which the oil to be tested flows into a receiving flask, Fig. 146, whose capacity to a mark on its neck is 60 (±0.15) c.c. The lower end of the outlet tube is enclosed by a larger tube, which, when stoppered by a cork, *C*, acts as a closed air chamber and prevents the flow of oil through the outlet tube until the cork is removed and the test started. A looped string may be attached to the lower end of the cork as an aid to its rapid removal. The temperatures in the standard oil tube and in the bath are shown by thermometers. The bath may be heated by any suitable means. The standard oil tube shall be thoroughly cleaned, and all oil entering the standard oil tube shall be strained through a 60-mesh wire

strainer. A stop watch shall be used for taking the time of flow of the oil and a pipette shall be used for draining the overflow cup of the standard oil tube.

(*b*) The standard oil tube, which may be standardized by the U. S. Bureau of Standards, Washington, D. C., shall conform to the following dimensions:

Dimensions	Minimum, cm.	Normal, cm.	Maximum, cm.
Inside diameter of outlet tube	0.1750	0.1765	0.1780
Length of outlet tube	1.215	1.225	1.235
Height of overflow rim above bottom of outlet tube	12.40	12.50	12.60
Diameter of container of standard oil tube	2.955	2 975	2.995
Outer diameter of outlet tube at lower end	0.28	0.30	0.32

3. Viscosity shall be determined at 100°F. (37.8°C.), 130°F. (54.4°C.), or 210°F. (98.9°C.). The bath shall be held constant within 0.25°F. (0.14°C.), at such a temperature as will maintain the desired temperature in the standard oil tube. For viscosity determinations at 100° and 130°F., oil or water may be used as the bath liquid. For viscosity determinations at 210°F., oil shall be used as the bath liquid. Viscosity determinations shall be made in a room free from draughts, and from rapid changes in temperature. All oil introduced into the standard oil tube, either for cleaning or for test, shall first be passed through the strainer.

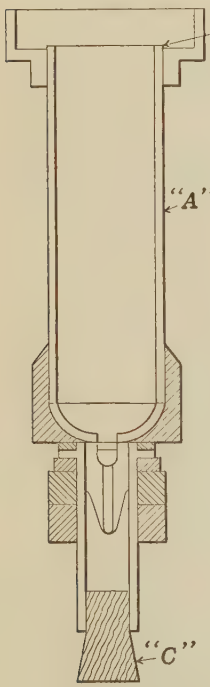


FIG. 145.—Sectional view of standard oil tube.

To make the test, heat the oil to the necessary temperature and clean out the standard oil tube. Pour some of the oil to be tested through the cleaned tube. Insert the cork stopper into the lower end of the air chamber at the bottom of the standard oil tube, sufficiently to prevent the escape of air, but not to touch the small outlet tube of the standard oil tube.

Heat the oil to be tested, outside the viscosimeter, to slightly below the temperature at which the viscosity

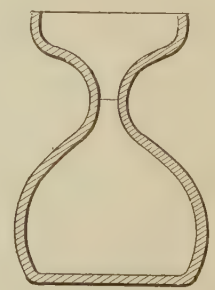


FIG. 146.—Sectional view of receiving flask.

is to be determined and pour it into the standard oil tube until it ceases to overflow into the overflow cup. By means of the oil tube thermometer keep the oil in the standard oil tube well stirred and also stir well the oil in the bath. It is extremely important that the temperature of the bath be maintained constant during the entire time consumed in making the test. When the temperatures of the bath and of the oil in the standard oil tube are constant, and the oil in the standard oil tube is at the desired

temperature, withdraw the oil tube thermometer; quickly remove the surplus oil from the overflow cup by means of a pipette so that the level of the oil in the overflow cup is below the level of the oil in the tube proper; place the 60-c.c. flask, Fig. 146, in position so that the stream of oil from the outlet tube will strike the neck of the flask so as to avoid foam. Snap the cork from its position, and at the same instant start the stop-watch. Stir the liquid in the bath during the run and carefully maintain it at the previously determined proper temperature. Stop the watch when the bottom of the meniscus of the oil reaches the mark on the neck of the receiving flask.

The time in seconds for the delivery of 60 c.c. of oil is the Saybolt viscosity of the oil at the temperature at which the test was made.

SPECIFICATIONS FOR LUBRICATING OILS

CLASS A

General:

1. This specification covers the grades of petroleum oil used by the United States Government and its agencies for the general lubrication of engines and machinery where a highly refined oil is not required. This oil is not to be used for steam-cylinder lubrication.

2. Only refined petroleum oils without the admixture of fatty oils, resins, soap, or other compounds not derived from crude petroleum, will be considered.

3. These oils shall be supplied in five grades, known as extra light, light, medium, heavy and extra heavy.

Properties and Tests:

4. Flash- and fire-points: The flash- and fire-points of the five grades shall not be lower than the following:

	Flash, °F.	Fire, °F.
Extra light	315	355
Light	325	365
Medium	335	380
Heavy	345	390
Extra heavy	355	400

5. Viscosity: The viscosity of the five grades of oil at 100°F. must be within the following limits:

	Seconds
Extra light	140–160
Light	175–210
Medium	275–310
Heavy	370–410
Extra heavy	470–520

6. Color: The color of the extra heavy grade shall not be darker than No. 6 National Petroleum Association Standard, or its equivalent. The color of the other grades shall not be darker than No. 5 National Petroleum Association Standard, or its equivalent.

7. Pour test: The pour test shall not be above the following temperatures:

	Degrees F.
Extra light	35
Light	35
Medium	40
Heavy	45
Extra heavy	50

8. Acidity: Not more than 0.10 milligram of potassium hydroxide shall be required to neutralize 1 gram of the oil.

9. Corrosion: A clean copper plate must not be discolored when submerged in the oil for 24 hours at room temperature.

10. All tests shall be made according to the methods adopted by the Committee on Standardization of Petroleum Specifications.

SPECIFICATIONS FOR LUBRICATING OILS
CLASS B

General:

1. This specification covers the grades of petroleum oil used by the United States Government and its agencies for the lubrication of turbines, dynamos, high-speed engines and other classes of machinery where an oil better than Class A is required. The oil must be satisfactory for use in circulating and forced-feed systems.

2. Only refined petroleum oils without the admixture of fatty oils, resins, soaps, or other compounds not derived from crude petroleum, will be considered.

3. These oils shall be supplied in five grades, known as extra light, light, medium, heavy and extra heavy.

Properties and Tests:

4. Flash- and fire-points: The flash- and fire-points of the five grades shall not be lower than the following:

	Flash, °F.	Fire, °F.
Extra light	315	355
Light	325	365
Medium	335	380
Heavy	345	390
Extra heavy	355	400

5. Viscosity: The viscosity of the five grades at 100°F. must be within the following limits:

	Seconds
Extra light	140–160
Light	175–210
Medium	275–310
Heavy	370–410
Extra heavy	470–520

6. Color: The color of the extra heavy grade shall not be darker than No. 6 National Petroleum Association Standard, or its equivalent. The color of the other grades shall not be darker than No. 5 National Petroleum Association Standard, or its equivalent.

7. Pour test: The pour test shall not be above the following temperatures:

	Degrees F.
Extra light	35
Light	35
Medium	40
Heavy	45
Extra heavy	50

8. Acidity: Not more than 0.07 milligram of potassium hydroxide shall be required to neutralize 1 gram of oil.

9. Corrosion: A clean copper plate must not be discolored when submerged in the oil for 24 hours at room temperature.

10. Emulsifying properties: The oil shall separate completely in 30 minutes from an emulsion with:

(1) Distilled water. (2) 1 per cent. salt solution. (3) Normal caustic soda solution.

The demulsibility shall not be less than 300.

11. All tests shall be made according to the methods adopted by the Committee on Standardization of Petroleum Specifications.

SPECIFICATIONS FOR LUBRICATING OILS
CLASS C

General:

1. This specification covers the grades of petroleum oil used by the United States Government and its agencies for lubrication of air compressors and internal combustion engines, except aircraft, motorcycle and Diesel engines; also for the lubrication of turbines and

other machinery where an oil better than Class B is required. This oil must be satisfactory for use in circulating and forced-feed systems.

2. Only refined petroleum oils without the admixture of fatty oils, resins, soaps, or other compounds not derived from crude petroleum, will be considered.

3. These oils shall be supplied in five grades, known as extra light, light, medium, heavy and extra heavy.

Properties and Tests:

4. Flash- and fire-points: The flash- and fire-points of the five grades shall not be lower than the following:

	Flash, °F.	Fire, °F.
Extra light	315	355
Light	325	365
Medium	335	380
Heavy	345	390
Extra heavy	355	400

Oil for use in air compressors where the air leaving any stage or cylinder has a temperature above 212°F. shall have a flash-point not lower than 400°F.

5. Viscosity: The viscosity of the five grades at 100°F. must be within the following limits:

	Seconds
Extra light	140–160
Light	175–210
Medium	275–310
Heavy	370–420
Extra heavy	470–520

6. Color: The color of the extra heavy grade shall not be darker than No. 6 National Petroleum Association Standard, or its equivalent. The color of the other grades shall not be darker than No. 5 National Petroleum Association Standard, or its equivalent.

7. Pour test: The pour test shall not be above the following temperatures:

	Degrees F.
Extra light	35
Light	35
Medium	40
Heavy	45
Extra heavy	50

8. Acidity: Not more than 0.05 milligram of potassium hydroxide shall be required to neutralize one gram of the oil.

9. Corrosion: A clean copper plate must not be discolored when submerged in the oil for 24 hours at room temperature.

10. Emulsifying properties: The oil shall separate completely in 30 minutes from an emulsion with:

(1) Distilled water. (2) 1 per cent. salt solution. (3) Normal caustic soda solution.

The demulsibility shall not be less than 300.

11. Carbon residue: The carbon residue shall not exceed the following:

	Per cent.
Extra light	0.10
Light	0.20
Medium	0.30
Heavy	0.40
Extra heavy	0.60

12. Further tests on oils of Class C may be required at the option of the Department of the Government using the oils.

13. All tests shall be made according to the methods adopted by the Committee on Standardization of Petroleum Specifications.

AIRCRAFT MACHINE GUN OIL

General:

1. This specification covers the grade of petroleum oil used by the United States Government and its agencies for the lubrication of machine guns on aircraft for the c.c. interrupter gears and for gun oil for cleaning and oiling machine guns and small arms.

2. The oil must be a highly refined, filtered, straight-run petroleum oil, suitable in every way for the uses specified in Paragraph 1. It must be a pure petroleum product, without the addition of vegetable or animal oils or fats of any kind. It shall not contain any material which might gum or corrode metals under any conditions.

Properties and Tests:

3. Flash-point: The flash-point shall not be less than 200°F.

4. Viscosity: The viscosity at 100°F. shall be within the following limits: 70 to 95 seconds.

5. Pour test: The pour test shall be 45 degrees or more below zero Fahr.

6. Acidity: Not more than 0.03 milligram of potassium hydroxide shall be required to neutralize 1 gram of oil.

7. Carbon residue: The carbon residue must not be more than 0.03 per cent.

8. All tests shall be made according to the methods adopted by the Committee on Standardization of Petroleum Specifications.

BUFFER OIL FOR RECOIL AND RECUPERATOR CYLINDERS OF ALL BRITISH TYPES OF HOWITZERS AND GUN CARRIAGES

General:

1. This specification covers the grade of petroleum oil used by the United States Government and its agencies for filling the recoil and recuperator cylinders of all British type howitzers and gun carriages.

2. The oil is to be a pure refined petroleum oil.

Properties and Tests:

3. The flash-point shall not be lower than 265°F.

4. Viscosity: The viscosity at 100°F. shall be within the following limits: 65 to 75 seconds.

5. Pour test: The pour test shall not be above 0°F.

6. Acidity: Not more than 0.05 milligram of potassium hydroxide shall be required to neutralize 1 gram of the oil.

7. All tests shall be made according to the methods adopted by the Committee on Standardization of Petroleum Specifications.

CUP GREASE

General:

1. This specification covers the grades of cup grease used by the United States Government and its agencies for the lubrication of such parts of motor equipment and other machinery as are lubricated by means of compression cups; No. ½ and No. 1 to be used in spindle cups or transmissions.

2. The grease must be a well-manufactured product, composed of a calcium soap and a highly refined mineral oil.

Properties and Tests:

3. The mineral oil used in reducing the soaps must be a straight well refined mineral oil, with a viscosity at 100°F. of not less than 100 seconds.

4. Soap base: This base to be a whole fat, such as pure tallow oil, neatsfoot oil, lard oil, horse oil, or other pure animal oil, used singly or in combination.

(*a*) No. ½ cup grease shall contain approximately 13 per cent. of a calcium soap made from an approved fat.

(*b*) No. 1 cup grease shall contain approximately 14 per cent. of a calcium soap made from an approved fat.

(*c*) No. 3 cup grease shall contain approximately 18 per cent. of a calcium soap made from an approved fat.

(*d*) No. 5 cup grease shall contain approximately 24 per cent. of a calcium soap made from an approved fat.

5. Consistency: These greases must be similar in consistency to the approved trade standards for No. ½, No. 1, No. 3, and No. 5 grease.

6. Moisture: The grease must be a boiled grease, containing not less than one or more than three per cent. of water when finished.

7. Corrosion: A clean copper plate must not be discolored when submerged in the grease for 24 hours at room temperature.

8. Ash: No. ½ grease—the ash shall not be greater than 1.7 per cent.
No. 1 grease—the ash shall not be greater than 1.8 per cent.
No. 3 grease—the ash shall not be greater than 2.3 per cent.
No. 5 grease—the ash shall not be greater than 3.5 per cent.

9. Fillers: The grease shall contain no fillers, such as resin, resinous oils, soapstone, wax, talc, powdered mica or graphite, sulphur, clay, asbestos, or any other filler.

10. All tests shall be made according to the methods adopted by the Committee on Standardization of Petroleum Specifications.[1]

TRANSMISSION LUBRICANT

General:

1. This specification covers the grade of petroleum oil used by the United States Government and its agencies for the lubrication of transmission gears and bearings, differential gears, worm drives, winch drives, and roller and ball bearings used in connection with such parts of the equipment of motor vehicles.

2. The lubricant must be a refined petroleum product, without the addition of any vegetable or animal oils or products derived from them, and be entirely free from fillers.

Properties and Tests:

3. Flash-point: The flash-point shall not be lower than 460°F.

4. Viscosity: The viscosity at 210°F. must be within the following limits: 175 to 220 seconds.

5. All tests shall be made according to the methods adopted by the Committee on Standardization of Petroleum Specifications.

MINERAL STEAM-CYLINDER OIL FOR NON-CONDENSING ENGINES

General:

1. This specification covers the grade of petroleum oil used by the United States Government and its agencies for non-condensing steam-engine cylinder lubrication where a mineral oil is required; also as a stock oil for compounding.

Properties and Tests:

2. The oil must be a well refined petroleum oil without compounding of any nature.

3. Flash-point: The flash-point must not be less than 475°F.

4. Viscosity: The viscosity at 210°F. must be within the following limits:

135 to 165 seconds.

5. Cold test: The cold test must not be above 45°F.

6. Precipitation test: When 5 c.c. of the oil are mixed with 95 c.c. of petroleum ether and allowed to stand 24 hours, it shall not show a precipitate or sediment of more than 0.25 c.c. (5 per cent. by volume of the original oil).

7. All tests shall be made according to the methods adopted by the Committee on Standardization of Petroleum Specifications.

COMPOUND STEAM-CYLINDER OIL FOR NON-CONDENSING ENGINES

General:

1. This specification covers the grade of petroleum oil used by the United States Government and its agencies for the lubrication of steam-cylinders of non-condensing engines and pumps where a compounded oil is required.

Properties and Tests:

2. The oil must be a well refined petroleum oil, compounded with not less than 5 nor more than 7 per cent. of acidless tallow oil or lard oil.

3. Flash-point: The flash-point must not be less than 475°F.

4. Viscosity: The viscosity at 210°F., must be within the following limits:

120 to 150 seconds.

5. Cold test: The cold test must not be above 45°F.

6. Precipitation test: When 5 c.c. of the oil are mixed with 95 c.c. of petroleum ether and

allowed to stand 24 hours, it shall not show a precipitate or sediment of more than 0.25 c.c. (5 per cent. by volume of the original oil).

7. Acidity: The oil must not contain more than 0.40 per cent. of acid calculated as oleic acid (equivalent to 0.80 mg. KOH per gram of oil).

8. All tests shall be made according to the methods adopted by the Committee on Standardization of Petroleum Specifications.

FLOOR OIL

General:

1. This specification covers the grade of oil used by the United States Government and its agencies for polishing and preserving wooden floors.

2. The oil must be a well refined straight petroleum oil.

Properties and Tests:

3. Flash-point: The flash-point shall not be less than 300°F.

4. Viscosity: The viscosity at 100°F. shall be within the following limits:

60 to 100 seconds.

5. Pour test: The pour test shall not be greater than 35°F.

6. Color: The oil shall be pale or red in color. Black oil will not be accepted.

7. All tests shall be made according to the methods adopted by the Committee on Standardization of Petroleum Specifications.

GEAR CHAIN AND WIRE ROPE LUBRICANT

General:

1. This specification covers the grade of petroleum oil used by the United States Government and its agencies for the lubrication and protection of chains, wire ropes and gears of cranes, dredges, steam shovels and all other heavy equipment, for the lubrication and protection of the gears and ropes of balloon hoists; and for swabbing the wires and cables of aircraft.

2. The oil must be a petroleum product only, free from vegetable or animal oils or products derived from them. It must be entirely free from fillers, such as talc, resin, and all materials of every nature not related to the original product.

Properties and Tests:

3. Viscosity: The viscosity at 210°F. must be within the following limits:

900 to 1100 seconds.

4. Protection: When applied to a plate of polished steel, the lubricant must protect the steel for a period of thirty days when immersed in a 10 per cent. salt solution.

5. All tests shall be made according to the methods adopted by the Committee on Standardization of Petroleum Specifications.

GUN AND ICE MACHINE OIL

General:

1. This specification covers the grades of petroleum oil used by the United States Government and its agencies for cleaning and oiling guns and small arms where aircraft machine gun oil is not required; also for lubrication of the cylinders of ice machines; for lubrication of pneumatic tools and for hydraulic systems.

2. The oil must be a straight-run, highly refined petroleum oil, free from vegetable or animal oils or products derived from them; must be suitable in every way for the uses listed in Paragraph 1; and must not gum or corrode metals under any conditions.

3. These oils shall be supplied in two grades known as No. 100 and No. 125.

Properties and Tests:

4. Flash-point: The flash-point must not be less than 300°F.

5. Viscosity: The viscosity at 100°F. must be within the following limits:

No. 100 oil	95 to 110 seconds.
No. 125 oil	120 to 135 seconds.

6. Pour test: The pour test shall not be higher than 5° above zero F.

7. Acidity: Not more than 0.03 milligram of potassium hydroxide shall be required to neutralize 1 gram of the oil.

8. Emulsifying properties: The oil shall separate completely in 30 minutes from an emulsion with:

(1) Distilled water.

(2) 1 per cent. salt solution.

(3) Normal caustic soda solution.

The demulsibility shall not be less than 300.

9. All tests shall be made according to the methods adopted by the Committee on Standardization of Petroleum Specifications.

HYDROLINE OIL

General:

1. This specification covers the grade of petroleum oil used by the United States Government and its agencies to fill the recoil cylinders of gun carriages.

2. The oil must be entirely neutral and free from acid or alkali, and from ash and saponifiable oil.

Properties and Tests:

3. Viscosity: The viscosity shall be not greater than 145 seconds at 32°F. and not less than 43 seconds at 100°F.

4. Pour test: The pour test must be below 0°F.

5. Evaporation test: The oil must not lose more than 5 per cent. in weight when heated at 212°F. for two hours. Preference will be given to oil having the lowest percentage loss in weight, other things being equal.

6. All tests shall be made according to the methods adopted by the Committee on Standardization of Petroleum Specifications.

LIBERTY AERO AND MOTORCYCLE OIL

General:

1. This specification covers the grade of petroleum oil used by the United States Government and its agencies for the lubrication of stationary-cylinder aircraft engines and motorcycle engines.

2. The oil must be made from pure, highly refined petroleum products and must be suitable in every way for the entire lubrication of stationary-cylinder aircraft engines and motorcycle engines operating under all conditions. The oil must not contain moisture, sulphonates, soap, resin, or tarry constituents which would indicate adulteration or lack of proper refining.

Properties and Tests:

3. Flash-point: The flash-point shall not be lower than 400°F.

4. Viscosity: The viscosity of the oil at 210°F. shall be within the following limits:

80 to 90 seconds.

5. Pour test: The pour test for Summer Oil shall not be above 45°F. For Winter Oil not above 20°F.

6. Acidity: Not more than 0.10 milligram of potassium hydroxide shall be required to neutralize 1 gram of the oil.

7. Emulsifying properties: The oil shall separate completely in one hour from an emulsion with:

(1) Distilled water. (2) 1 per cent. salt solution.

At a temperature of 180°F.

8. Carbon residue: The carbon residue shall not be over 1.5 per cent.

9. All tests shall be made according to the methods adopted by the Committee on Standardization of Petroleum Specifications.

MARINE ENGINE OIL

General:

1. This specification covers the grade of oil used by the United States Government and its agencies for the lubrication of reciprocating steam engines in marine service where a compounded engine oil is required.

THIS OIL MUST NOT BE USED IN CIRCULATING OR FORCED-FEED SYSTEMS.

Properties and Tests:

2. The oil shall be a compounded oil made from refined petroleum oil and 10 per cent. to 20 per cent. of blown refined rape seed oil or blown refined peanut oil; so compounded that it will not separate or break down in any way, either before or while in service.

3. Viscosity: The viscosity shall be: 65 to 75 seconds at 210°F. Not over 700 seconds at 100°F.

4. Pour test: The pour test shall not be above 32°F.

5. Acidity: The oil shall not contain more than 1.50 per cent. of acid calculated as oleic acid (equivalent to 3.0 mg. KOH per gram of oil).

6. Corrosion: A clean copper plate must not be discolored when submerged in the oil for 24 hours at room temperature.

7. Emulsifying properties: The oil shall remain completely emulsified for an hour from an emulsion with:

(1) Distilled water. (2) 1 per cent. salt solution.

8. Wick feed: The oil shall show a flow at the end of 14 days of at least 30 per cent. of its flow at the end of the first 24-hour period.

9. All tests shall be made according to the methods adopted by the Committee on Standardization of Petroleum Specifications.

OIL AND GREASE USED IN RECOIL MECHANISM OF 75 AND 155 MM. GUN CARRIAGE (FRENCH)

General:

1. This specification covers the grade of petroleum oil and grease used by the United States Government and its agencies for the recoil mechanism of 75 and 155 mm. French gun carriages.

Recuperator Oil:

2. This oil shall be a highly refined petroleum product, free from animal or vegetable oils.

Properties and Tests:

3. Flash-point: The flash-point shall not be lower than 345°F.

4. Viscosity: The viscosity at 100°F. shall be within the following limits:

385 to 430 seconds.

5. Pour test: The pour test shall be 5 or more degrees below zero F.

6. Acidity: Not more than 0.05 milligram of potassium hydroxide shall be required to neutralize 1 gram of the oil.

7. Corrosion: A clean copper plate must not be discolored when submerged in the oil for 24 hours at room temperature.

Recuperator Grease:

8. The grease must be a well manufactured product composed of a calcium soap and a highly refined mineral oil.

Properties and Tests:

9. The mineral oil used in reducing the soap must have a viscosity at 100°F. of not less than 180 seconds.

10. Soap base: The grease shall contain approximately 18 per cent. of a calcium soap made from a whole fat, such as pure tallow oil, neatsfoot oil, lard oil, horse oil, or other pure animal oil, used singly or in combination.

11. Consistency: This grease must be similar in consistency to the approved trade standard for No. 3 grease.

12. Moisture: The grease must be a boiled grease containing not less than 1 nor more than 3 per cent. of water when finished.

13. Corrosion: A clean copper plate must not be discolored when submerged in the grease for 24 hours at room temperature.

14. Ash: The ash shall not be greater than 2.3 per cent.

15. Fillers: The grease shall contain no fillers, such as rosin, resinous oils, soapstone wax, talc, powdered mica or graphite, sulphur, clay, asbestos or any other filler.

16. All tests shall be made according to the methods adopted by the Committee on Standardization of Petroleum Specifications.

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HORACE BINNEY.

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